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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

SQ-14J

MEMORANDUM

DATE: MAR 29 1995

SUBJECT: Review of the Draft PRP-Lead Quality Assurance Project Plan (QAPP) for Central Support Zone Investigation at the Enviro-Chem Superfund Site, Zionsville, Indiana

FROM: George Schupp, Chief
Quality Assurance Section

TO: Bruce Sypniewski, Chief
Section 2, Remedial Response Branch

Attention: Dion Novak, Remedial Project Manager (RPM)

We have reviewed the subject QAPP for Central Support Zone Investigation at the Enviro-Chem Superfund Site, Zionsville, Indiana. The subject QAPP, which was received by the Quality Assurance Section (QAS) on March 10, 1995 (QAS SF Log-in No.2155) is not acceptable. The attached comments itemize QAPP deficiencies and provide guidance for their correction. If there are any questions regarding this memorandum, the RPM can contact Ida Levin of my staff.

Attachment

cc: K. Khanna, HSRLT-5J

ATTACHMENT

Review of the Draft PRP-Lead Quality Assurance Project Plan (QAPP) for Central Support Zone Investigation at the Enviro-Chem Superfund Site, Zionsville, Indiana

I. PROJECT DESCRIPTION

A. It should be explained/referenced in Section 1.6, last paragraph on page 1-6, how the samples for full laboratory analysis will be selected.

B. The Data Quality Objective (DQO) Levels used for this project should be more specific. Use attached Summary Table 1 as an example.

C. Table 1-5 (Site-Specific Acceptable Soil Concentrations for VOCs) was not referenced in any section of the QAPP. It should be explained whether these high site-specific acceptable soil concentrations provided for the clean up levels.

II. PROJECT ORGANIZATION AND RESPONSIBILITIES

Due to reorganization of the ESD the references to the Central Regional Laboratory (CRL), Laboratory Scientific Support Section (LSSS) in Section 2.10 and Figure 2-1 should be changed to Monitoring and Quality Assurance Branch (MQAB), Contract Analytical Services Section (CASS).

III. QUALITY ASSURANCE OBJECTIVIES FOR MEASUREMENT DATA

It should be expected for the superfund project to meet the QC acceptance criteria for 95 percent or more for all samples tested using the CLP SOW. This statement should be added in Section 3.3 for completeness.

IV. SAMPLING PROCEDURES

A. The "Specification and Guidance for Contaminant-Free Sample Containers, **December 1992**" should be used in Appendix D.

B. In Section 3.2 of Field Sampling Plan soil samples for screening will be collected in 8-ounce glass jar, while in the Appendix B, SOP 25, Section 25.4.1, page 3 of 4, samples for headspace analysis will be collected in the plastic bag. Please correct the discrepancy.

C. Soil samples for VOC analyses should be collected in two 120-ml VOA vials. This statement should be added to the sampling plan.

V. CALIBRATION PROCEDURES AND FREQUENCY

In Section 6.2 the CLP SOW OLM01.8 should be referenced for calibration of the laboratory equipment, not Ceimic Corporation Laboratory QA Plan.

VI. ANALYTICAL PROCEDURES

A. Section 7.2. There is no need to reference SOP, if CLP SOW OLM01.8 will be used for the VOC analyses.

B. Comments will not be provided on Appendix B (SOP for VOCs), due to use of CLP SOW OLM01.8.

VII. INTERNAL QUALITY CONTROL CHECKS

In Section 8.3 CLP SOW OLM01.8 should be referenced for Internal QC check in the laboratory.

VIII. DATA REDUCTION, VALIDATION AND REPORTING

A. In Section 9.1.2 CLP SOW OLM01.8 should be referenced for laboratory data reduction.

B. "U.S.EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, **February 1994**" should be used for data validation in Section 9.2.

C. Section 9.3. We strongly recommend to use the CLP reporting format.

IX. PERFORMANCE AND SYSTEM AUDITS

Section 10.2. The external field and Laboratory audits will be performed by Region 5, Monitoring and Quality Assurance Branch (MQAB), Contract Analytical Services Section (CASS).

X. PREVENTIVE MAINTENANCE

An example of the table showing the type of maintenance to be performed (field and laboratory) and the frequency is appropriate. The referenced Section 12 of Appendix C does not provide these information. Use the Superfund Model QAPP as a guideline.

LEVELS OF DATA QUALITY OBJECTIVES

Sample Matrix	Field Parameters	Laboratory Parameters	Level of DQOs
TAILINGS	Radio-activity, Seismic	--	I
		Soil Physical Testing	III
		Asbestos	V
		CLP RAS Metals	IV
		Extractable Organics	IV
SEDIMENT		Soil Physical Testing	III
		Asbestos	V
		CLP RAS Metals	IV
		Extractable Organics	IV
SURFACE WATER	pH, Conductivity, D.O.	--	I
		Water Indicators	V
		CLP RAS Metals	IV
		Extractable Organics	IV
		Asbestos	V
GROUNDWATER	pH, Conductivity, D.O.	--	I
		Resistivity	I
		CLP RAS Metals	IV
		Water Indicator Parameter	V
Soil		Asbestos	V
		CLP RAS Metals	IV
		Extractable Organics	IV

DRAFT

CENTRAL SUPPORT ZONE INVESTIGATION QUALITY ASSURANCE PROJECT PLAN

ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, INDIANA

PREPARED FOR

ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION TRUST

PREPARED BY

DOW ENVIRONMENTAL INC. PITTSBURGH, PENNSYLVANIA

DEI PROJECT NUMBER 2455.003

MARCH 1995

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QUALITY ASSURANCE PROJECT PLAN (QAPP)

CENTRAL SUPPORT ZONE INVESTIGATION
ENVIRO-CHEM SUPERFUND SITE
ZIONSVILLE, INDIANA

MARCH 1995

PREPARED BY:

DOW ENVIRONMENTAL INC.
PITTSBURGH, PENNSYLVANIA

APPROVALS: DATE:

_____ _____
ECC TRUST'S REPRESENTATIVE (PRP) (OPTIONAL)

_____ _____
REMEDIAL CONTRACTOR PROJECT MANAGER

_____ _____
INDEPENDENT CONSTRUCTION QUALITY ASSURANCE OFFICER

_____ _____
U.S. EPA REGION V, REMEDIAL PROJECT MANAGER

_____ _____
U.S. EPA REGION V, QUALITY ASSURANCE MANAGER

_____ _____
IDEM PROJECT COORDINATOR

INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been developed to cover all anticipated chemical and physical parameter testing which will be conducted during the Environmental Conservation and Chemical Corporation Site (ECC Site) Central Support Zone Investigation (SZI) at the ECC Site located in Zionsville, Indiana.

This QAPP is organized per the U.S. EPA Region V Office of RCRA, "Model Quality Assurance Project Plan" May 1991 as well as the U.S. EPA Region V "Contents of Laboratory Standard Operating Procedures", 1989. Ceimic Corporation of Pittsburgh, Pennsylvania has been utilized for purposes of satisfying the associate laboratory QA sections of the QAPP. Ceimic Corporation has not been approved by the ECC Trust or U.S. EPA/IDEM however. Section 4.0 per the "Model QAPP" calls for sampling procedures which are presented in Section 3.0, and in a number of Standard Operating Procedures (SOPs) included in Appendix B of the SZI Field Sampling Plan prepared separately by Dow Environmental Inc. (DEI). Appendix B contains information on the selected laboratory for this project in terms of operating procedures for the stated methodology and general laboratory practices.

1.0 PROJECT DESCRIPTION

The following is a description of historical information on the site in terms of location, collected data, current project scope and objective, sampling approach, chemical constituents to be tested for, project data objectives, and schedule.

1.1 Site Location and Background Information

The ECC Site is located in a rural area of Boone County, about 5 miles north of Zionsville and 10 miles northwest of Indianapolis, Indiana (Figures 1-1 and 1-2). The site is defined as the area bounded by the perimeter fence, constructed during the Site Preparation and Material Removal (SPMR) activities conducted in 1993. This area includes the 3.053-acre remedial boundary, the support zone, and a buffer zone between the fence and the north and eastern sides of the site.

Directly west of the site is an active commercial waste handling and recycling facility operated by the Boone County Resource Recovery Systems, Inc. (BCRRS). Access to the site will be from State Route 421 and will be within a property easement shared with BCRRS.

Directly east of the site across an unnamed ditch is the inactive Northside Sanitary Landfill (NSL) Landfill. This facility is also a Superfund Site and is presently undergoing remedial design activities. The south end of the site is approximately 500 feet from an existing residence and is approximately 400 feet from Finley Creek, the main surface water drainage in the site area.

Residential properties are also located to the north and west, within 1/2 mile of the facilities. A small residential community, Northfield, is located north of the site on State Route 421. Approximately 50 residences are located within 1 mile of the site.

The site is in an area that is gently sloping, predominantly to the east towards the unnamed ditch. The unnamed ditch runs north to south along the eastern edge of the site and drains the site either directly or from tributary ditches on the north and south ends of the site. The unnamed ditch flows south from the site to Finley Creek. During the SPMR activities, diversion ditches were to be constructed in order to handle and direct storm water to the unnamed ditch.

These ditches were not fully completed due to soils being encountered which exhibited elevated organic vapor meter (OVM) readings during excavation of the south support zone diversion channel near the center area of the site in the vicinity of the old access road. The findings were reported to the U.S. EPA and detailed in the SPMR Monthly Progress Reports covering the period between September 11, 1993 and November 11, 1993. The location of the area is represented by a hatch area on Figure 1-3 and occurs in a gravel layer approximately 1 foot below grade. This layer is approximately 1/2-foot thick and was exposed during the excavation activities to a length of approximately 5 feet. After this layer was identified, the diversion channel excavation efforts in this location were stopped, the excavated channel was backfilled.

1.2 Previous Data Collection and Current Requirements

The Enviro-Chem Trustee's Engineer (the Engineer) notified the U.S. EPA and the Indiana Department of Environmental Management (IDEM) and prepared the "Sampling and Analysis Plan for Diversion Channel Northwest of Concrete Pad" (the Hand Auger Investigation Plan) which was submitted to IDEM and the U.S. EPA on behalf of the Enviro-Chem Trustees on October 12, 1993. The U.S. EPA verbally provided comments on the plan on October 13. The Hand Auger Investigation Plan included soil boring by hand auger in the area of potential contamination, field screening by headspace analysis with an OVM, and laboratory analysis for Volatile Organic Compounds (VOCs) of selected soil samples. The field sampling was initiated on October 13, 1993.

The results of the field screening activities were reported in the SPMR Monthly Progress Report Number 3. The screening and sampling locations, HA-1 through HA-20, are shown on Figure 1-4. Table 1-1 presents a summary of the field screening results. Subsurface conditions prevented retrieval of samples from below the 0.5 to 1.5 depth in certain locations, therefore, areal distribution of detectable VOCs could not be reliably estimated by field screening. Additionally, one soil sample in the diversion channel area (support zone sample) and two samples west of the area (HA-3 and HA-16) were selected for laboratory analysis of VOCs by U.S. EPA's SW-846 Method 8240A. The sampling locations were selected to establish the soil concentrations at increasing distances west of the location of potential contamination and the remedial boundary. Analytical results of the support zone sample (1.0 feet) and samples HA-3 (0.5 to 1.0 foot) and HA-16 (1.5 to 2.0 feet) are summarized on Table 1-2. Certificates of Analysis are included in Appendix A. The analytical results indicate that the concentrations of VOCs decrease with increasing distance west of the remedial boundary. Table 1-3 shows the

comparison of VOC results of the HA-3 and HA-16 to criteria outlined in the Consent Decree which defines the acceptable soil VOC constituent concentrations for soil cleanup verification based upon their mean concentration from a group of samples, no VOC constituent exceeded established acceptable soil concentrations.

1.3 Project Scope and Objectives of Support Zone Investigation

The central support zone area occurs along the boundary of the site in the vicinity of the former truck access road into the facility. The three soil samples collected at this location revealed the greatest concentration in the ditch along the western boundary of the site with decreasing concentrations in the support zone westward from the ditch. This area is relatively limited in size and the mean concentration of the samples does not exceed acceptable concentrations for any VOC constituent. However, additional soil sampling is proposed in this area to determine the extent of VOC constituents in proximity to the site and to obtain a more representative sample group to evaluate cleanup levels.

Soil samples will be taken during the support zone investigation to assess the horizontal and vertical (to 10 feet) extent of volatile organic compounds (VOCs) in the area of the central support zone.

1.4 Sampling Approach Rationale

For the Central Support Zone Area, the distance between soil borings was determined by using the formula included in Instructions for the Preparation of Closure Plans for Interim Status Facilities by the Illinois Environmental Protection Agency (IEPA) - Division of Land Pollution Control, dated March 2, 1989. The formula is as follows:

$$GI = (A/\pi)^{0.5}/2$$

where:

GI	=	Grid interval in feet
A	=	Area in square feet

For this calculation, the area of this investigation was estimated to be the limits of the field screening investigation as described in Section 1.1.1 which is approximately 100 x 150 feet, or 15,000 square feet. Using this as the total sampling area, a soil boring spacing interval of 35 feet is obtained. Based on this estimate a grid spacing of 50 feet was selected based on the knowledge that sampling beyond the proposed grid will only proceed in a west to southwest direction if required because of the existing Remedial Action Boundary on the north and east sides of the grid. Spacing can be reduced, if appropriate, by drilling in between the proposed spacing if contaminant concentrations exceeded acceptable levels.

Twelve test borings will be drilled on a 50-foot spaced grid to obtain subsurface soil samples for VOC analysis in the central support zone area. Proposed soil boring locations are shown on Figure 1-5. Each borehole will be drilled utilizing hollow stem augers. Samples will be obtained continuously on 2-foot intervals to a depth of 10 feet by split-barrel samplers. Each sample will be screened utilizing a photoionization detector (PID) to determine gross VOC concentration. In addition to the screened sample, a portion of each sample obtained will also be placed in a laboratory VOC sample jar in the event that the sample will be selected and submitted for laboratory analysis.

The initial selection criteria to be applied will be sample depth. This criteria will ensure that adequate vertical delineation of the area is achieved. The second criteria for laboratory sample selection will be field screening results. At a minimum, two samples from each borehole will be submitted for VOC analysis. One sample will be submitted from the 0- to 5-foot interval and a second sample will be submitted from the 5- to 10-foot interval. Additional samples may be taken from a boring based on field conditions (e.g., a drastic change in soil classification and headspace readings at the end of a 5-foot interval where insufficient soil is available for sampling).

If laboratory analysis results for any of the west or south perimeter grid sample points exceed the acceptable soil concentration values in Table 1-3, the grid will be expanded further in that direction along the gridline for a distance of 50 feet, or less. All additional grid sample borings will have a minimum of two soil samples submitted for VOC analysis.

1.5 Analytical Testing

1.5.1 Field Screening

A portion of each soil sample will be submitted for field screening utilizing real-time measurement based on photoionization detection. The sample shall be representative of the candidate laboratory sample, which will also be collected from the split spoon at the time of sample retrieval. If two distinctly different soil types are encountered within a sample, each portion will be screened separately assuming an adequate sample volume is available.

There are potentially 60 samples, five from each 10-foot boring (one per each 24-inch split spoon barrel) that will be field screened.

1.5.2 Laboratory Analysis

Soil samples will be submitted to Ceimic Corporation of Pittsburgh, Pennsylvania for confirmatory analysis of TCL VOCs by using the most recent U.S. EPA-approved version of CLP SOW OLM01.8. The requirements for precision, accuracy, completeness, representativeness, and comparability are described in Section 3.0 for both field and laboratory testing.

The collection of samples including quality assurance/quality control (QA/QC) volumes are summarized in Table 1-4.

1.6 Data Quality Objectives and Intended Data Uses

DQOs are qualitative and quantitative statements defined by U.S. EPA that specify the quality of the data required to support decisions made during site remediation activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels that address various data uses and the QA/QC efforts and methods required to achieve the desired level of quality. These levels are:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at a site, preliminary comparison of site data to Applicable or Relevant and Appropriate Requirements (ARARs), initial site characterization to locate areas that require subsequent and more accurate analyses, and engineering screening of alternatives (bench-scale tests).

- Field Analyses (DQO Level 2): This provides rapid results and better quality than Level 1 analyses. This level may include mobile laboratory-generated data depending on the level of quality control exercised.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile laboratory-generated data and some analytical laboratory methods (e.g., laboratory data with quick turnaround used for screening but without full QC documentation).

- Conformational (DQO Level 4): This provides the highest level of data quality and is used for the purposes of conducting a risk assessment, evaluating remedial alternatives, and determining the Potentially Responsible Parties. These analyses require full Contract Laboratory Program (CLP) analytical methods and data validation procedures in accordance with U.S. EPA-recognized protocols.

- Nonstandard (DQO Level 5): This refers to analyses by nonstandard protocols, for example, when exact detection limits or the analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data.

The primary data uses for the ECC Site SZI sampling are to provide, through real-time field screening (DQO Level 1) measurements, results to adjust levels of protection to personnel involved with drilling and sampling and to support decision making on selection of appropriate samples for full laboratory analysis as per Section 1.5.2.

The laboratory analysis of samples, selected through the field screening process and based on the criteria of headspace readings (generally the highest recorded sample value from the upper and lower 5-foot intervals of the 10-foot boring) and depth of sampling, will be conducted under the highest level (DQO Level 4) of data quality using an U.S. EPA-approved method utilized by the CLP for the determination of TCL VOCs.

A minimum of 10 percent of the offsite samples will represent nondetectable headspace values in order to quantify the entire VOC concentration range in samples collected.

1.7 Project Schedule

The Central Support Zone Investigation will start 2 weeks after U.S. EPA approval of the Field Sampling Plan and Quality Assurance Project Plan. Field work is expected to be completed within 2 weeks. A tentative start date of May 16, 1995 has been presented to U.S. EPA in the draft project schedule, submitted on March 7, 1995.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The U.S. EPA and IDEM will be responsible for the government reviews associated with the SZI. The ECC Trust has the overall responsibility for implementing required work at the site. DEI as the Remedial Design Engineer for the ECC Site has prepared the Field Sampling Plan (FSP) and this QAPP. The SZI Contractor(s) will be DEI who will be responsible for implementation of investigative activities based on the requirements of this QAPP.

The various QA and management responsibilities of key project personnel associated with environmental sampling and analyses are defined in the following subsections. A project organization chart, which includes the lines of authority, is included as Figure 2-1.

2.1 ECC Trust

The ECC Trust will have the overall responsibility for the implementation of the SZI. The ECC Trust and/or their designee have the authority to commit the resources necessary to meet the project objectives and requirements.

The ECC Trust will: (1) provide the major point of contact with the U.S. EPA and IDEM for matters concerning the project; (2) ensure that the project activities meet the requirements of the Consent Decree; and (3) approve all external reports (deliverables) before their submission to the Agencies.

2.2 U.S. EPA Remedial Project Manager

The U.S. EPA Remedial Project Manager (RPM), will be responsible for overseeing the project and coordinating the U.S. EPA and IDEM's review and approval of this document and associated plans for the SZI.

2.3 IDEM Remedial Project Manager

The IDEM RPM will be responsible for overseeing the project and for conducting all IDEM reviews of the SZI associated plans.

2.4 SZI Contractor Project Manager

The ECC Trust will select DEI as the SZI Contractor(s) to perform the investigation. The Contractor Project Manager will have the overall responsibility for ensuring that the project meets the objectives and the quality standards specified in this QAPP.

The Contractor Project Manager will: (1) acquire and apply technical resources as needed to ensure performance within budget and schedule constraints; (2) orient, direct, and monitor the field staff; (3) review the work performed and data obtained to ensure its quality, responsiveness, and timeliness; and (4) be responsible for the preparation and quality of subsequent reports submitted to the Agencies.

2.5 DEI Site Manager

DEI' Site Manager will be responsible for leading and coordinating the day-to-day activities of the drilling crew and the Field Sample Custodian. The Site Manager will be an experienced geologist and will report directly to the Project Manager. Specific responsibilities will include: (1) implementation of the Field Sampling Plan; (2) acquisition and documentation of subsurface data; (3) coordination with and assistance to the Field Sample Custodian; (4) compliance with QA/QC requirements described in this QAPP; (5) compliance with the corrective action procedures described in this QAPP; and (6) participation in the preparation of the final report.

2.6 SZI Contractor Field Sample Custodian

The Contractor's Field Sample Custodian will be responsible for the collection, screening, onsite custody, and packaging and shipping of all required samples as directed by the geologist and requirements set forth by this QAPP. Additionally, the Field Sample Custodian will be responsible for associated sample documentation and coordination with and providing direction to the selected laboratory.

2.7 U.S. EPA Region V Quality Assurance Officer

The U.S. EPA Region V QAO will have the responsibility of reviewing and approving this QAPP.

2.8 Subcontract Laboratory Project Manager

The analyses to be performed by the laboratory subcontractor is listed in Section 1.5.2. Ceimic Corporation has been selected by the SZI Contractor as the project laboratory and will be approved by the ECC Trust and U.S. EPA/IDEM. The laboratory Project Manager will be responsible for coordinating and scheduling the laboratory analyses; supervising the in-house chain of custody; accepting requirements outlined within this QAPP; and overseeing the data review and preparation of the analytical reports.

2.9 Subcontract Laboratory Quality Assurance Officer

The laboratories' QAOs will be responsible for overseeing the laboratory QA and the analytical results QA/QC documentation, conducting the data review, selecting any necessary laboratory corrective actions, adherence to applicable in-house SOPs, adherence to the QAPP, and approving the final analytical reports. The laboratory may have more than one QAO if, for example, any of these various activities take place in different departments within the laboratory.

2.10 U.S. EPA Region V Central Regional Laboratory

The Laboratory Scientific Support Section of the Central Regional Laboratory (CRL) of U.S. EPA Region V will be responsible for external performance and system audits of the analytical laboratory.

3.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objective is to develop and implement procedures for sampling, chain of custody, laboratory analyses, field measurements, and reporting that will provide data of a quality consistent with its intended use and remain defensible in a court of law. Specific procedures for sampling, chain of custody, laboratory and field instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of equipment, and corrective action are described in other sections of this QAPP. This section addresses the accuracy, precision, sensitivity, completeness, representativeness, and comparability of analyses.

3.1 Level of Quality Control Effort

Field duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field duplicate samples are analyzed to check for sampling reproducibility. Matrix spikes (MS) provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes for organic analyses are performed in duplicate and are hereinafter referred to as MS/MSD samples.

The general level of QC effort will include one field duplicate for every 10 or fewer investigative samples.

The general level of QC effort will also include one MS/MSD analysis for every 20 or fewer samples. For organics in soil samples designated for MS/MSD analysis, no extra sample volume is needed. The number of MS/MSD and duplicate field samples to be collected are listed in Table 1-4. Sampling procedures are specified in the FSP.

The levels of QC effort by the selected analytical laboratory is detailed in their SOP (see Appendix B).

3.2 Accuracy, Precision, and Sensitivity of Analyses

The QA objectives of laboratory analyses with respect to accuracy, precision, and sensitivity are to achieve the QC acceptance criteria of the analytical protocols. Accuracy and precision requirements for CLP protocol analyses are described in SOW OLM01.8. Examples of accuracy

and precision criteria for volatiles in soil are described in the SOP in Appendix B. These parameters are defined and assured by Ceimic Corporation in Section 4.0 of their Laboratory QA Plan provided in Appendix C.

The QA objectives for the field screening surveys conducted using real-time measuring instruments are to obtain reliable results of potential volatile organic vapors as headspace in order to make field decisions for selection of samples for full-scale laboratory testing. Accuracy, Precision, and Sensitivity of Analyses using real-time instruments is maintained by following the procedures of operation specified by the manufacturer's instrumentation manual in regard to operation, calibration, and maintenance.

3.3 Completeness, Representativeness, and Comparability

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the laboratory will provide data which will supply sufficient information to make logical decisions concerning positioning of potential additional required borings and field sampling.

Representativeness expresses the degree to which data accurately and precision represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper selection of laboratory protocols. This sampling and analysis program is designed to provide data representative of the subsurface conditions which are existing in the Central Support Zone area. The sampling procedures which are specified in the FSP were developed giving special consideration to existing analytical results from the Hand Auger Investigation, the physical characteristics of the materials and debris, and the potential need for future action in this area. Representativeness will be achieved using proper sampling and handling techniques (specified in this QAPP and the FSP), by properly preserving the samples, extracting and analyzing the samples within the required holding times, and using clean and appropriate sample containers (see Appendix D). The adequacy of the sampling procedures will be assessed by analyzing field duplicates.

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as described in the QAPP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of differences in procedures, QA objectives, and media being tested.

These parameters are also defined and assured by Ceimic Corporation in Section 4.0 of their Laboratory QA Plan (see Appendix C).

4.0 SAMPLING PROCEDURES

Sampling procedures for the CSI are provided in Section 3.0 and Appendix B (SOPs) of the SZI FSP prepared by DEI (February 1995).

5.0 SAMPLE CUSTODY PROCEDURES

This QAPP presents the sample custody protocols described in "NEIDC Policies and Procedures" (EPA-330/9-78-DDI-R, revised June 1985). Sample custody consists of three parts: sample collection, laboratory analysis, and final evidence files. A sample or evidence file will be considered under a person's custody if it: (1) is in a person's physical possession, (2) is in view of the person after he/she has taken possession, (3) has been secured by that person so that no one can tamper with the sample, or (4) has been secured by that person in an area that is restricted to authorized personnel. Final evidence files, including all originals of laboratory reports and field files, will be maintained in a secure area.

5.1 Field Chain-of-Custody Procedures

The field sampling and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain of custody intact. The protocols for specific sample numbering are included in Section 4.1 of the FSP. A copy of the chain-of-custody form is provided in SOP Number 9 (see Appendix B) in the FSP.

5.1.1 Field Procedure

The field custody procedures to be followed by all sampling personnel include:

- The Field Sample Custodian will be personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples.

- All samples will be tagged with sample numbers and locations.

- Sample tags will be completed for each sample using waterproof ink unless prohibited by weather conditions.

5.1.2 Field Logbooks/Documentation

Field logbooks will provide the means of documenting the activities performed at the Site. As such, entries will be in as much detail as possible so that persons going to the Site could reconstruct a particular situation without relying on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by a project-specific number.

The title page of each logbook will contain the following information:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- End date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the Site and field sampling or investigation team personnel as well as the purpose of their visit will also be recorded in the field logbook.

All measurements will be recorded and all of the collected samples will be described in the field logbook. All entries will be made in ink, and no erasures will be permitted. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is taken, a detailed description of the location, which includes compass and distance measurements, shall be recorded. The numbers of the photographs taken of the location, if any, will also be noted. All equipment used to take measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures specified in the FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, and volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field QA/QC samples, which will receive entirely separate sample identification numbers, will be noted under the sample description.

5.1.3 Transfer-of-Custody and Shipment Procedures

The transfer-of-custody and shipment procedures will be as follows:

- Samples will be accompanied by a properly completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the transfer of custody of samples from the sampler to another person, to a permanent laboratory, or to/from a secure storage area.

- Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. Custody seals will be attached to the front right and back left of the cooler and will be covered with clear plastic tape. The cooler will be strapped shut with strapping tape in at least two locations.

- The original chain-of-custody record and the yellow and pink copies will accompany the shipment. The gold copy will be retained by the samplers and returned to the field office.

5.2 Laboratory Chain-of-Custody Procedures

The specifications for chain-of-custody and document control for Ceimic Corporation are described in Section Number 6 of their Laboratory Quality Assurance Plan (see Appendix C).

5.3 Final Evidence Files Custody Procedures

DEI will maintain the SZI evidence files until instructed to turn them over to the ECC Trust or his representative. The evidence files will include all relevant records, correspondence, reports, logs, field logbooks, laboratory sample preparation and analysis forms, data packages, pictures, subcontractor reports, chain-of-custody records, and data review reports. The evidence files will be under the custody of the DEI Project Manager in a secure area.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes the procedures for maintaining the accuracy of all the instruments and measuring equipment that are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled, periodic basis.

6.1 Field Instruments/Equipment

Instruments and equipment used to gather, generate, or measure chemical parameters of interest will be calibrated with sufficient frequency and in such a manner to ensure that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used during the field sampling will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments will include an Organic Vapor Meter (OVM) with Photoionization Detection (PID).

6.2 Laboratory Equipment

Calibration of laboratory equipment will be based on written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing QC activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a repair staff with in-house spare parts or will maintain service contracts with vendors.

For the analyses conducted for samples selected and submitted during the SZI, the calibration procedures and frequencies specified in Ceimic Corporation's Laboratory QA Plan, Section Number 7, as provided in Appendix B of this QAPP will be utilized.

7.0 ANALYTICAL PROCEDURES

7.1 Field Screening by Headspace Analysis

DEI SOP Number 25 presented in Appendix B of the FSP presents a field procedure for acquiring real-time measurement of VOCs by means of a soil sample headspace vapor technique.

7.2 Laboratory Analysis

The U.S. EPA Contract Laboratory Statement of Work (SOW) OLM01.8 will be utilized for the determination of Target Compound List (TCL) Volatile Organic Compounds (VOCs). Ceimic Corporation's Standard Operating Procedure (SOP) Number 004 covers this methodology and is provided in Appendix B of this QAPP.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Field Sample Collection

All the field QC will be carried out in accordance with the procedures described in this QAPP. Field QC will include:

- Appropriate sample collection, as per DEI SOP Number 12 in Appendix B of the FSP.
- Proper decontamination of sampling equipment after each use, as described in DEI SOP Number 8 (Appendix B) and Section 3.4 of the FSP.
- Proper calibration of the field instruments, as established in Section 6.1 of this QAPP.

8.2 Field Measurements

QA for field measurements will consist of review of OVM calibration of the field instrument and replication of measurements to ensure reproducibility as specified in DEI SOP Number 25 provided in Appendix B of the FSP.

8.3 Laboratory Analyses

Ceimic Corporation will implement a QA program and QC checks to ensure the generation of analytical data of known and documented usable quality. This is outlined in Section 10.0 of their Laboratory QA Plan provided in Appendix C of this QAPP.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

Procedures for documenting sample collection and custody, validating analytical data, and reporting the results of the SZI are covered in this section.

9.1 Data Reduction

9.1.1 Field Measurements and Sample Collection

Field measurements and sample collection data will be recorded in the field logbook. If these data are to be used in the project reports, they will be reduced and summarized, and the method of reduction will be documented in the specific report. Sample custody and analysis requests will be documented on chain-of-custody records.

9.1.2 Laboratory Services

Analytical data reduction will be carried out by each laboratory performing analysis on the soil samples. The data reduction will be reviewed and checked as part of the data evaluation and decision making process for soil boring placement beyond the proposed grid. Compounds detected in blanks will not be subtracted from analytical results of investigative samples and will be reported separately.

9.2 Data Validation

Data validation will consist of review and evaluation of field and/or laboratory QA/QC sample data by DEI's Data Validation and Risk Assessment Group in Pittsburgh, Pennsylvania using the following:

- U.S. EPA "National Functional Guidelines for Organic Data Review," dated June 1991.
- U.S. EPA Region V "Standard Operating Procedure for Validation of CLP Organic Data," dated April 1991.

- An assessment of whether the samples were properly collected and handled according to the Field Sampling Plan (FSP) and Section 5.0 of this QAPP.

- A check on received results against chain-of-custody records to determine completeness.

- A check on the comparability of field duplicates.

- Review of internal laboratory QA/QC as outlined within SOW OLM01.8 and Ceimic Corporation's SOP Number 004.

- An evaluation of the laboratory's ability to meet quality control criteria for:

    - Initial and continuing calibrations
    - Spiked sample results (surrogates, matrix spikes, LCS samples)
    - Comparability of laboratory duplicates
    - Evaluation of laboratory method blank results
    - Correct compound identification
    - Proper compound quantitation
    - Correct transcription of analytical results

Ceimic Corporation will perform in-house analytical data validation under direction of each laboratory's QAO as follows:

- The laboratory will check for the attainment of QC criteria as outlined in the their SOP Number 004.

- The laboratory will assess the validity of analytical data by comparing the analytical results of duplicate, MS/MSD, and laboratory blank samples.

- The laboratory will critique their own analytical programs by using spiked addition recoveries, established detection limits, and precision and accuracy control charts and by keeping accurate records of calibrations.

9.3 Reporting

Reporting of chemical results on selected soil boring samples will include the following:

- Cover sheets listing the samples included in the report
- Tabulated results on soils analyzed for TCL VOC parameters
- Analytical results for QC sample spikes and sample duplicates

Section Number 9 of Ceimic Corporation's Laboratory QA Plan covers data reduction, review, and reporting and is provided in Appendix C of this QAPP.

10.0 PERFORMANCE AND SYSTEM AUDITS

The Remedial Contractor Quality Control Manager for the ECC Site will monitor and audit the performance of QA/QC procedures to ensure that the SPMR activities are executed in accordance with the FSP and this QAPP.

10.1 Field Activities

DEI's Site Manager will monitor and audit the performance of field QA/QC procedures by reviewing the detailed description of sample collection and field measurement procedures recorded in the field notebook to ensure that this investigation is executed in accordance with this FSP.

The field audits will include an evaluation of sampling methods; sample handling and packaging; equipment use; equipment decontamination, maintenance, and calibration procedures; and chain-of-custody procedures. In addition, all records and documentation procedures will be reviewed to ensure compliance with the project requirements. Any deviations from the FSP or the QAPP will be recorded in the field notebook by the person conducting the audit, who will then inform the personnel involved in the activity of the problem and notify the Project Manager for initiation of any necessary corrective action procedures.

10.2 Laboratory

Ceimic Corporation shall complete their own internal procedural and system audits as presented in Section Number 11 of their Laboratory QA Plan provided in Appendix C of this QAPP. Ceimic Corporation has been informed that the Validation Manager, representatives of U.S. EPA Region V Central Regional Laboratory, and IDEM reserve the right to perform independent audits at any period of time before, during, and after the project activities.

11.0 PREVENTATIVE MAINTENANCE

11.1 Field Equipment

Preventative maintenance procedures for field equipment will be those recommended by the manufacturers. Field instruments will be checked and calibrated by the supplier prior to shipment and in the field as described in Section 6.1.

Critical spare parts will be kept onsite to minimize instrument down time. Back-up equipment will be available by 1-day shipment.

11.2 Laboratory Equipment

As part of their QA/QC program, Ceimic Corporation performs routine preventative maintenance to minimize the occurrence of instrument failure and other system malfunctions and have a designated internal group who are responsible for performing routine scheduled maintenance and repairing or coordinating the repair of all instruments with the appropriate vendor. All laboratory instruments are maintained in accordance with the manufacturer's specifications and the requirements of the specific method being employed. This maintenance program is carried out on a regular, scheduled basis, and documented in the laboratory service logbook for each instrument. Information detailing the type of maintenance that was performed and the frequency is included in the Laboratory QA Plan, Section Number 12 provided in Appendix C of this QAPP.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Field Measurements

Field data will be assessed by the Site Manager, who will review the field calibration logs and frequency as specified in the FSP and this QAPP. The accuracy of field measurements will be evaluated by using daily instrument calibration, and calibration checks.

12.2 Laboratory Data

Laboratory results will be assessed for compliance with the required precision, accuracy, completeness, and sensitivity as described in the following subsections.

12.2.1 Precision

The precision of laboratory analyses will be assessed by comparing the analytical results between matrix spike (MS) samples for the organic analyses.

The relative percent difference (%RPD) will be calculated for each pair of duplicate analyses by using Equation 12-1:

$$\%RPD = \frac{S - D}{(S + D)/2} \times 100 \qquad \text{(Equation 12-1)}$$

where:

S = First sample value (original or MS value)
D = Second sample value (duplicate or MSD value)

12.2.2 Accuracy

The accuracy of laboratory results will be assessed by using the analytical results of method blanks, reagent/preparation blanks, and MS samples. The percent recovery (%R) of MS samples will be calculated using Equation 12-2:

$$\%R = \frac{A - B}{C} X\ 100 \qquad \text{(Equation 12-2)}$$

where:

A	=	The analyte concentration determined experimentally from the spiked sample
B	=	The background level determined by a separate analysis of the unspiked sample
C	=	The amount of the spike added

12.2.3 Completeness

The data completeness of laboratory analytical results will be assessed for compliance with the amount of data required for decision making. Data completeness will be calculated by using Equation 12-3:

$$\%\ Completeness = \frac{Useable\ Data\ Obtained}{Total\ Data\ Realized} x\ 100 \quad \text{(Equation 12-3)}$$

12.2.4 Sensitivity

The achievement of method detection limits depends on the instrument's sensitivity and matrix effects. Therefore, it is important to monitor the instrument's sensitivity to ensure the data quality through appropriate instrument performance. The instrument's sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples.

13.0 CORRECTIVE ACTION

Corrective actions may be required for two classes of problems: sampling and analytical problems and noncompliance problems. Sampling and analytical problems may occur or be identified during the collection, handling, or preparation of a sample; laboratory instrument analysis; and data review.

For problems of noncompliance with the QAPP or the FSP, a corrective action program will be defined in accordance with this QAPP and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Project Manager. Implementation of the corrective action will be confirmed through the same channels.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by a stop-work order from the U.S. EPA or IDEM.

13.1 Sample Collection/Field Measurements

Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformances, or suspected deficiencies of any activity or issued document by reporting the situation to the Project Manager. The Site Manager will discuss the suspected problems with the Project Manager and if necessary with the ECC Trust, who will then make a decision based on the potential for the situation to affect the quality of the data. If it is determined that the situation is a corrective action unresolvable in a reasonable amount of time or because of boundary constraints, the U.S. EPA and IDEM will be notified.

The Project Manager will be responsible for ensuring that any corrective action is initiated by:

- Evaluating all reported problems or discrepancies.
- Controlling additional work on problem resolution.

- Determining disposition or action to be taken, in consultation with the ECC Trust if necessary and, if warranted by the situation, with the U.S. EPA and IDEM.

- Maintaining a log of corrective actions.

- Reviewing any required reports and corrective actions taken.

- Ensuring that required reports, if any, are included in the final site documentation in project files.

If appropriate, the Project Manager will ensure that no additional work that is dependent on the activity in dispute is performed until the corrective actions are completed.

Corrective actions for field measurements may include:

- Repeating the measurement to check the error
- Checking batteries
- Checking the calibration of the instrument
- Recalibrating the instrument
- Replacing the instrument or measurement devices
- Stopping work (if necessary)

The Site Manager will be responsible for all site activities and may be required to adjust the site investigation to accommodate site-specific needs. When it becomes necessary to modify an approach, the Site Manager will notify the Project Manager of the anticipated change and will implement the necessary changes after obtaining any required approval. The change in SZI approach will be documented by the Site Manager. Any U.S. EPA and IDEM approval for the change will be determined prior to field implementation, if feasible. Otherwise, the action taken during the period of modification will be evaluated to determine the significance of any departure from previously approved and established investigative scope.

The Contractor's Project Manager is responsible for controlling, tracking, and implementing any required changes. Reports on all changes will be distributed to all affected parties, including the U.S. EPA and IDEM. The U.S. EPA and IDEM will be notified whenever SZI scope or procedural changes are made in the field.

13.2 Laboratory Analyses

Corrective actions at the laboratories will be required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken will be somewhat dependent on the analysis and the event. Laboratory personnel will be alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy.
- Blanks contain target analytes above acceptable levels.
- Undesirable trends are detected in spike recoveries or in the %RPD between duplicates or MS.
- Unusual changes in detection limits are identified.
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples if used.
- Inquiries concerning data quality are received.

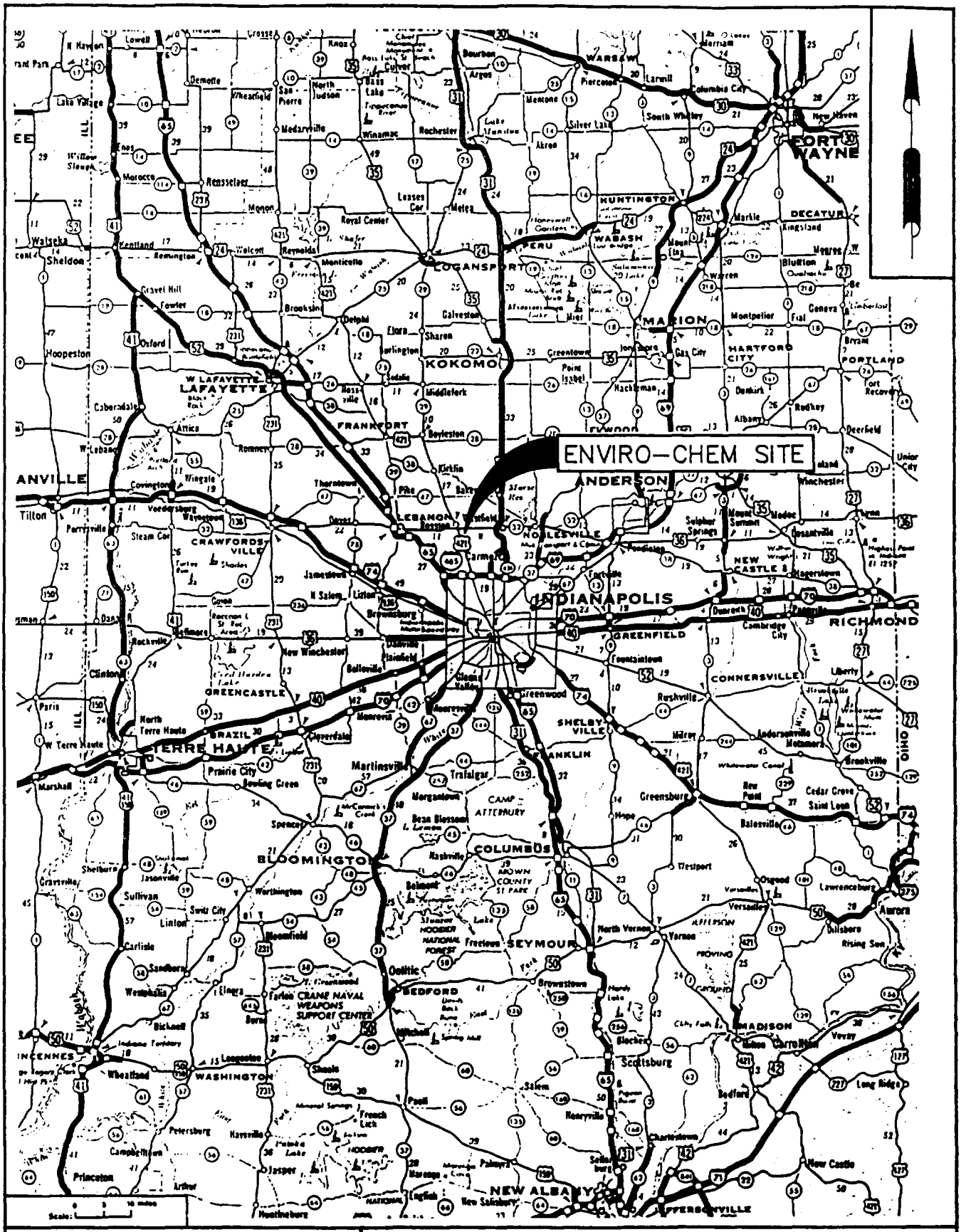
Corrective action procedures will often be handled at the bench level by the analyst, who will review the preparation or extraction procedure for possible errors; check the instrument calibration, spike and calibration mixes, and instrument sensitivity; and conduct other QA/QC reviews. If the problem persists or cannot be identified, the matter will be referred to the laboratory supervisor, Project Manager, and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the QA department. If the problem requires resampling or is not correctable in the laboratory, the laboratory QAO will notify DEI's Project Manager. The Project Manager will decide, in consultation with the ECC Trust and (if warranted by the significance of the problem) with the U.S. EPA and IDEM, the corrective actions to be implemented.

Ceimic Corporation has system corrective action steps outlined in Section Number 14 of their Laboratory QA Plan provided in Appendix C of this QAPP.

14.0 QUALITY ASSURANCE REPORTING

Quality Assurance reports will be issued by the Remedial Contractor. These documents will: (1) contain information that summarizes the QA activities in both the field and the laboratory, including audit results; (2) discuss any quality issues that required corrective action and document the corrective action that was taken; and (3) note any project problems that have occurred and any QA/QC issues that have been satisfactorily completed. Any problem serious enough to require significant actions (e.g., changing from an approved laboratory) will be reported to the U.S. EPA and IDEM within 5 days of the occurrence.

FIGURES



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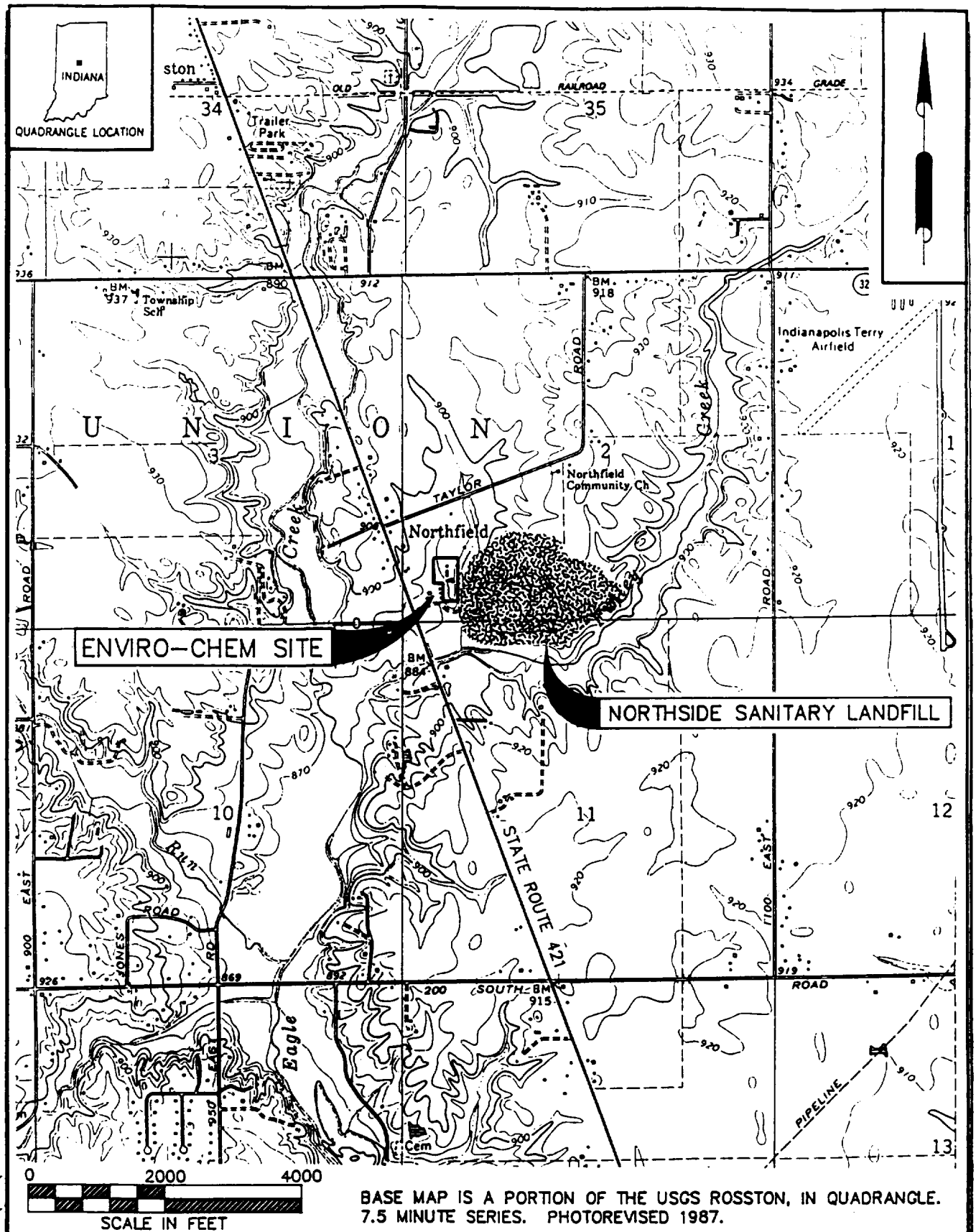
Dow Environmental

SITE LOCATION MAP

ENVIRO–CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NUMBER 2455–003

SCALE: AS SHOWN — FIGURE NUMBER 1–1 — REV 0



BASE MAP IS A PORTION OF THE USGS ROSSTON, IN QUADRANGLE. 7.5 MINUTE SERIES. PHOTOREVISED 1987.

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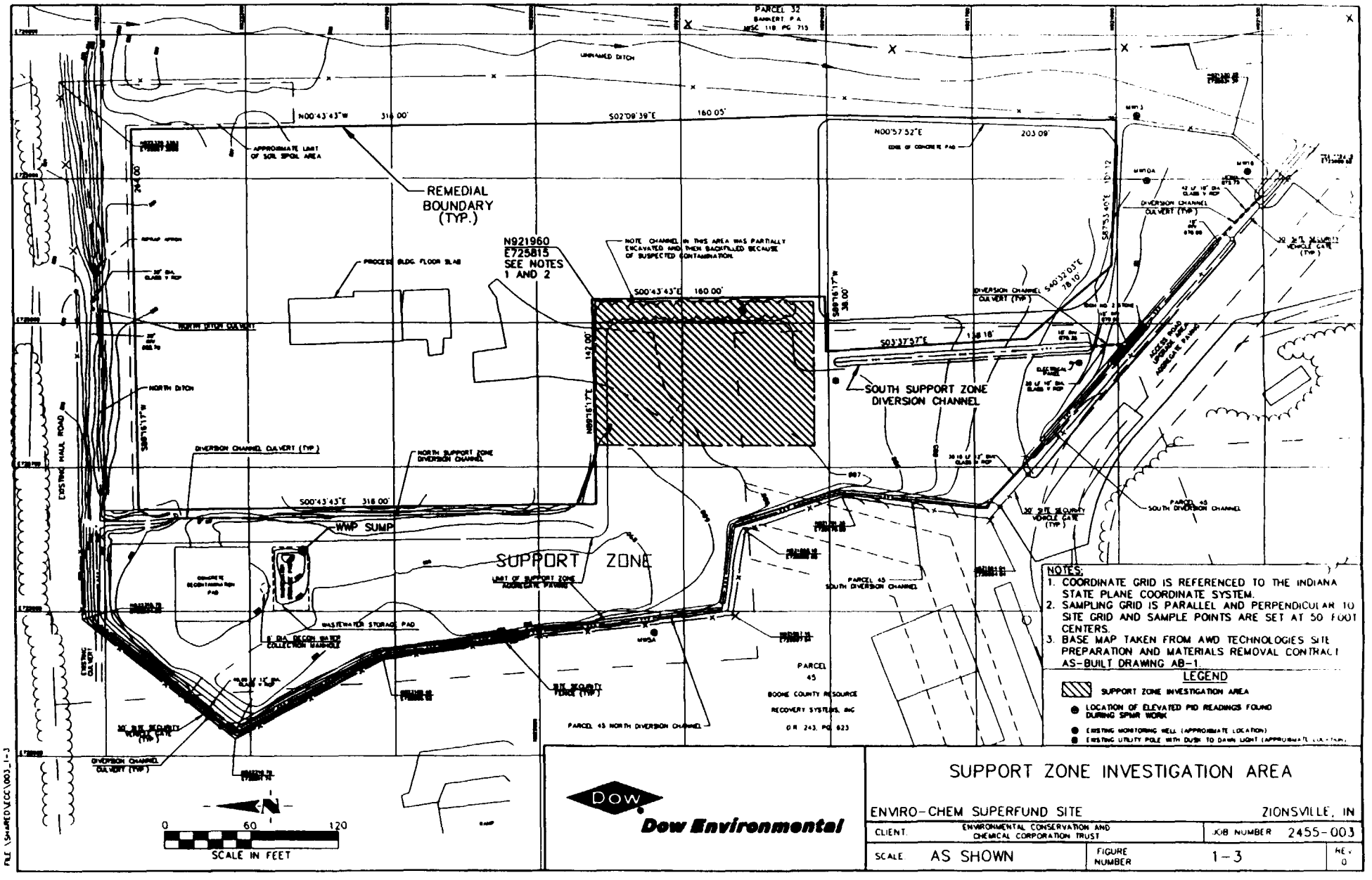
SITE MAP

ENVIRO–CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION & CHEMICAL CORP. TRUST — JOB NUMBER 2455–003

SCALE: AS SHOWN — FIGURE NUMBER 1–2 — REV 0



NOTES:

1. COORDINATE GRID IS REFERENCED TO THE INDIANA STATE PLANE COORDINATE SYSTEM.
2. SAMPLING GRID IS PARALLEL AND PERPENDICULAR TO SITE GRID AND SAMPLE POINTS ARE SET AT 50 FOOT CENTERS.
3. BASE MAP TAKEN FROM AWD TECHNOLOGIES SITE PREPARATION AND MATERIALS REMOVAL CONTRACT AS-BUILT DRAWING AB-1.

LEGEND

- SUPPORT ZONE INVESTIGATION AREA
- LOCATION OF ELEVATED PID READINGS FOUND DURING SPWP WORK
- EXISTING MONITORING WELL (APPROXIMATE LOCATION)
- EXISTING UTILITY POLE WITH DUSK TO DAWN LIGHT (APPROXIMATE LOCATION)

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Dow Environmental

SUPPORT ZONE INVESTIGATION AREA

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT	ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION TRUST	JOB NUMBER	2455-003
SCALE	AS SHOWN	FIGURE NUMBER	1-3
		REV	0

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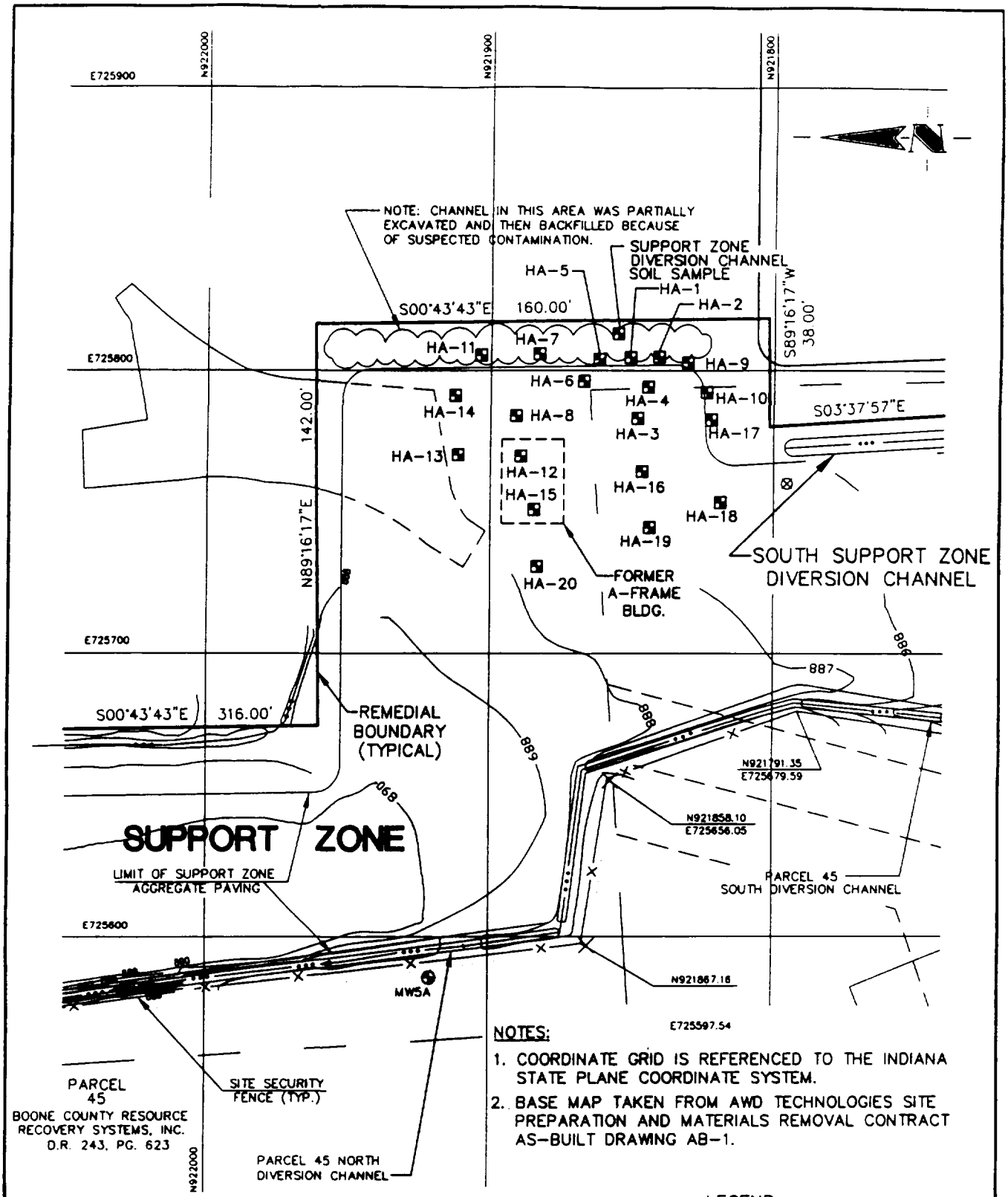

NOTES:

1. COORDINATE GRID IS REFERENCED TO THE INDIANA STATE PLANE COORDINATE SYSTEM.
2. BASE MAP TAKEN FROM AWD TECHNOLOGIES SITE PREPARATION AND MATERIALS REMOVAL CONTRACT AS-BUILT DRAWING AB-1.

LEGEND

HA-19 ■ HAND AUGER INVESTIGATION SOIL SAMPLE LOCATION

⊕ EXISTING MONITORING WELL (APPROXIMATE LOCATION)

⊗ EXISTING UTILITY POLE WITH DUSK TO DAWN LIGHT (APPROXIMATE LOCATION)

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0030001090 5RTH

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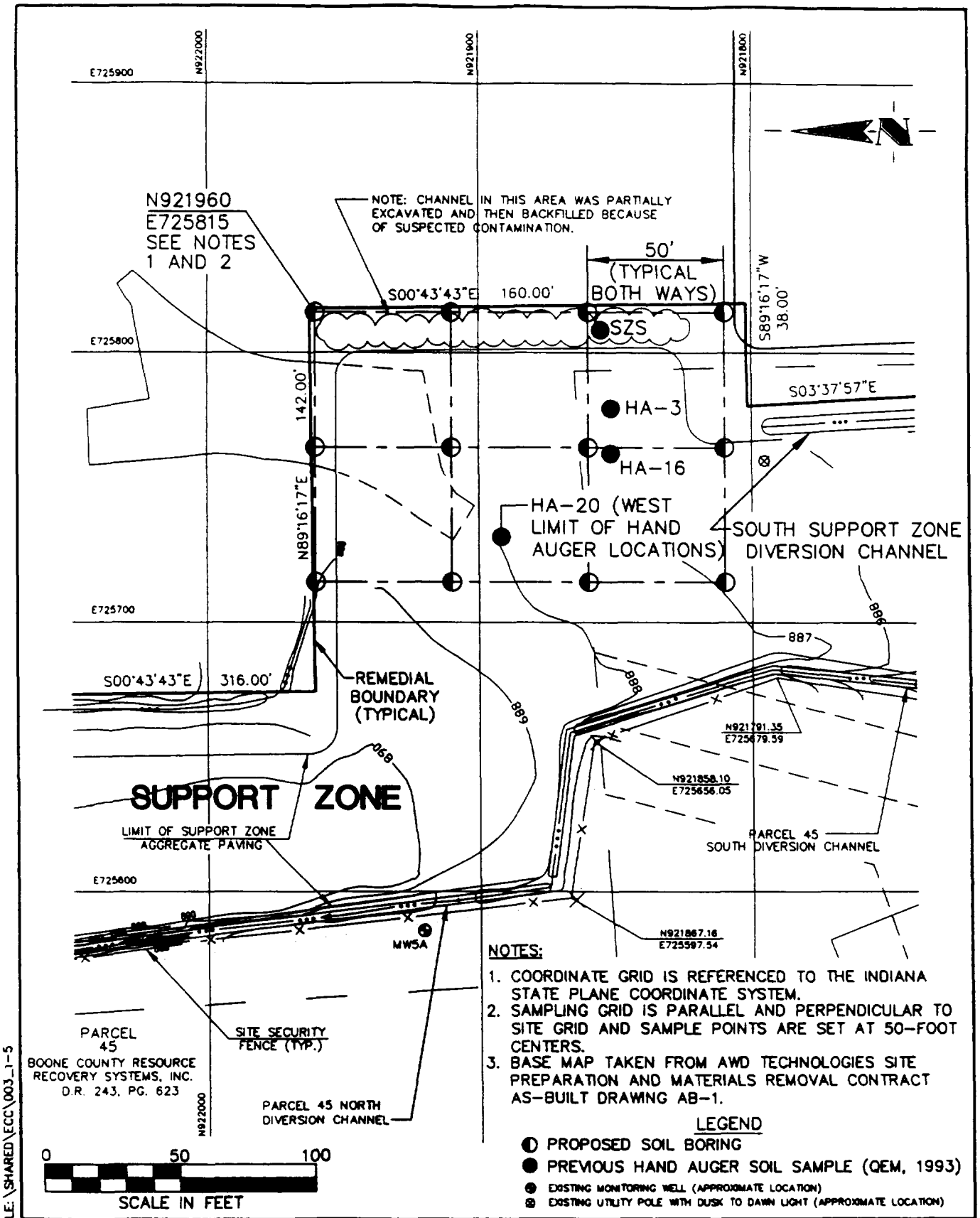
PREVIOUS SOIL SAMPLE LOCATIONS

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT:	ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION TRUST	JOB NUMBER:	2455-003
SCALE:	AS SHOWN	FIGURE NUMBER	1-4
		REV	0



NOTES:

1. COORDINATE GRID IS REFERENCED TO THE INDIANA STATE PLANE COORDINATE SYSTEM.
2. SAMPLING GRID IS PARALLEL AND PERPENDICULAR TO SITE GRID AND SAMPLE POINTS ARE SET AT 50-FOOT CENTERS.
3. BASE MAP TAKEN FROM AWD TECHNOLOGIES SITE PREPARATION AND MATERIALS REMOVAL CONTRACT AS-BUILT DRAWING AB-1.

LEGEND

- ◐ PROPOSED SOIL BORING
- ● PREVIOUS HAND AUGER SOIL SAMPLE (QEM, 1993)
- ⊕ EXISTING MONITORING WELL (APPROXIMATE LOCATION)
- ⊗ EXISTING UTILITY POLE WITH DUSK TO DAWN LIGHT (APPROXIMATE LOCATION)

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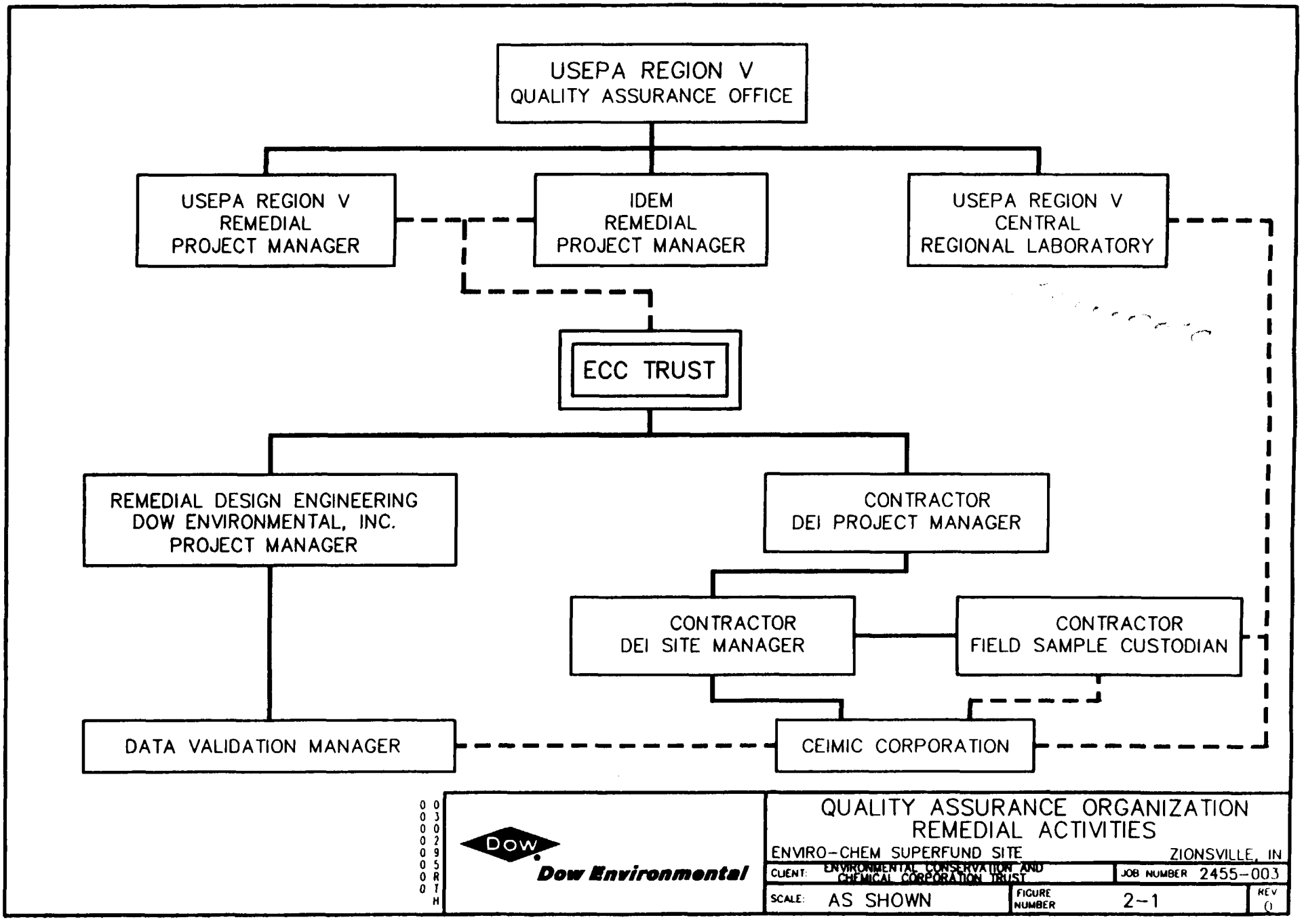
CENTRAL SUPPORT ZONE INVESTIGATIONS
SOIL SAMPLING GRID LOCATIONS

ENVIRO-CHEM SUPERFUND SITE — ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION TRUST	JOB NUMBER: 2455-003	
SCALE: AS SHOWN	FIGURE NUMBER 1-5	REV 0

FILE: \SHARED\ECC\003_2-1



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QUALITY ASSURANCE ORGANIZATION
REMEDIAL ACTIVITIES

ENVIRO-CHEM SUPERFUND SITE ZIONSVILLE, IN

CLIENT: ENVIRONMENTAL CONSERVATION AND CHEMICAL CORPORATION TRUST

JOB NUMBER 2455-003

SCALE: AS SHOWN

FIGURE NUMBER 2-1

REV 0

TABLES

TABLE 1-1

SOIL SAMPLING HEAD SPACE RESULTS
HAND AUGER INVESTIGATION (1)
ENVIROCHEM SITE
ZIONSVILLE, INDIANA

DEPTH BGS (feet)	HA-1 OVA READINGS	HA-2 OVA READINGS	HA-3 OVA READINGS	HA-4 OVA READINGS	HA-5 OVA READINGS	HA-6 OVA READINGS	HA-7 OVA READINGS	HA-8 OVA READINGS	HA-9 OVA READINGS	HA-10 OVA READINGS
0.0 - 0.5	10	24	1	0	7	50	40	>1,000	62	1
0.5 - 1.0	220	NS AR	1.5 S	0	>1,000	220	79	>1,000	9	0
1.0 - 1.5	NS AR	NS	NS	0	NS AR	NS AR	NS AR	NS AR	0	20
1.5 - 2.0	NS	NS	3	0.5	NS	NS	NS	NS	0	10
2.0 - 2.5	NS	NS	NS	0.5	NS	NS	NS	NS	1	34
2.5 - 3.0	NS	NS	16	0	NS	NS	NS	NS	3	50
3.0 - 3.5	NS	NS	NS	1.5	NS	NS	NS	NS	6	200
3.5 - 4.0	NS	NS	0.5	4.5	NS	NS	NS	NS	NS AR	850
4.0 - 4.5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS AR
4.5 - 5.0	NS	NS	1	NS	NS	NS	NS	NS	NS	NS

DEPTH BGS (feet)	HA-11 OVA READINGS	HA-12 OVA READINGS	HA-13 OVA READINGS	HA-14 OVA READINGS	HA-15 OVA READINGS	HA-16 OVA READINGS	HA-17 OVA READINGS	HA-18 OVA READINGS	HA-19 OVA READINGS	HA-20 OVA READINGS
0.0 - 0.5	2	4	NS AR	NS AR	23	15	55	58	600	34
0.5 - 1.0	NS AR	42	NS	NS	9	33	NS AR	14	>1000	97
1.0 - 1.5	NS	NS AR	NS	NS	NS AR	700	NS	NS AR	620	NS AR
1.5 - 2.0	NS	NS	NS	NS	NS	1000 S	NS	NS	400	NS
2.0 - 2.5	NS	NS	NS	NS	NS	300	NS	NS	NS AR	NS
2.5 - 3.0	NS	NS	NS	NS	NS	210	NS	NS	NS	NS
3.0 - 3.5	NS	NS	NS	NS	NS	280	NS	NS	NS	NS
3.5 - 4.0	NS	NS	NS	NS	NS	110	NS	NS	NS	NS
4.0 - 4.5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
4.5 - 5.0	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

Key:
OVA = Organic vapor analyzer.
NS = No sample.
BGS = Below ground surface.
S = Sample collected
AR = Auger refusal

Note:
(1) OVA headspace readings in Volumetric parts per million.

TABLE 1-2

SOIL SAMPLING ANALYTICAL RESULTS
HAND AUGER INVESTIGATION
ENVIROCHEM SITE
ZIONSVILLE, INDIANA

PARAMETERS	Acceptable Concentration (1)	Support Zone Sample	HA-3 0.5-1.0'	HA-16 1.5-2.0'
Volatile Organic Compounds (2)				
Methylene chloride	20	42	8	5
Toluene	238000	75	20	6
1,1,1-Trichloroethane	7200	710	74	11
Trichloroethene	240	73	21	7
Tetrachloroethene	130	320	ND	ND

Key:
HA = Hand auger sample.
ND = Not detected.

Notes:
(1) Reference: Table 3-1 Exhibit A Consent Decree.
(2) EPA Method (SW846-8240A) soil concentration in (ug/kg).

TABLE 1-3 COMPARISON OF WESTERN BOUNDARY RESULTS TO EXHIBIT A SOIL CLEANUP CRITERIA CENTRAL SUPPORT ZONE AREA			
PARAMETER	**CENTRAL SUPPORT ZONE AVERAGE**	**ACCEPTABLE CONCENTRATION**	**NUMBER OF SAMPLES**
METHYLENE CHLORIDE	18	25	3
TOLUENE	34	297500	3
1,1,1-TRICHLOROETHANE	265	9000	3
TRICHLOROETHENE	34	300	3
TETRACHLOROETHENE	108	163	3

NOTES:
ALL VALUES PROVIDED IN ug/kg .
THE ACCEPTABLE CONCENTRATION IS TAKEN FROM EXHIBIT A TABLE 3-1 AND FOOTNOTE 6 (TABLE 3-1 CONCENTRATIONS PLUS 25%)

TABLE 1-4

INITIAL TEST BORING SOILS TO BE COLLECTED FOR ANALYSIS
ECC SITE
CENTRAL SUPPORT ZONE INVESTIGATION

Sample Matrix	Field Parameter	Laboratory Parameter	Samples for Boring[1]	Field QA/QC Samples		
				Field Duplicates	MS/MSD Samples[2]	Total
Boring Soil	Total VOCs (PID)	TCL Volatiles	2 (5)	3 (3)	--- (NA)	27 (63)

Notes

(1) Twelve initial borings are proposed.

(2) For inorganics, organics in soil and soil vapor analyses, no extra volume is required.

TABLE 1-5 SITE-SPECIFIC ACCEPTABLE SOIL CONCENTRATIONS FOR VOCs	
Compounds	Acceptable Soil Concentration (μg/kg)
Acetone	490
Chlorobenzene	10,100
Chloroform	2,300
1,1-Dichloroethane	5.7
1,1-Dichloroethene	120
Ethylbenzene	234,000
Methylene Chloride	20
Methyl Ethyl Ketone	75
Methyl Isobutyl Ketone	8,900
Tetrachloroethene	130
Toluene	238,000
1,1,1-Trichloroethane	7,200
1,1,2-Trichloroethane	22
Trichloroethene	240
Total Xylenes	195,000
Vinyl Chloride	1.9
1,2-Dichloroethane	34.2
Dimethyl Phthalate	
Trichlorofluoromethane	864,703
1,2-Dichloroethene (Total)	514
2,4-Dimethylphenol	
1,2-Dichlorobenzene	349,807
Butyl Benzyl Phthalate	

APPENDIX A

CERTIFICATES OF ANALYSIS - HAND AUGER INVESTIGATION

CERTIFICATE OF ANALYSIS

Service Location	Received	Project	Lab ID
HERITAGE LABORATORIES, INC. 7901 W. MORRIS ST. INDIANAPOLIS, IN 46231 (317)243-8305	14-OCT-93	2506	A293253
	Complete: 03-NOV-93	PO Number:	
	Printed: 03-NOV-93	Sampled: 14-OCT-93 13:25	

Report To

ROBERT J. AUTIO
QUALITY ENVIRONMENTAL MANAGEMENT
1640 STRICKLAND
MARTINSVILLE, IN 46151

Bill To

CHARLES JACKSON
QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555
ROCKVILLE, IN 47872

Sample Description

DESCRIPTION: SUPP. ZONE DIVERSION CHANNEL

VOLATILE ORGANICS (HEATED PURGE & TRAP) SW846-8240A

Analyst: S. MAZUR Analysis Date: 26-OCT-93 12:13 Instrument: GC/MS VOA Test: 0510.9.0

Parameter	Result	Det. Limit	Units
ACETONE	BDL	100	ug/kg
ACROLEIN	BDL	250	ug/kg
ACRYLONITRILE	BDL	350	ug/kg
BENZENE	BDL	25	ug/kg
BROMODICHLOROMETHANE	BDL	25	ug/kg
BROMOFORM	BDL	25	ug/kg
BROMOMETHANE	BDL	50	ug/kg
CARBON DISULFIDE	BDL	25	ug/kg
CARBON TETRACHLORIDE	BDL	25	ug/kg
CHLOROBENZENE	BDL	25	ug/kg
CHLOROETHANE	BDL	50	ug/kg
CHLOROFORM	BDL	25	ug/kg
CHLOROMETHANE	BDL	50	ug/kg
DIBROMOCHLOROMETHANE	BDL	25	ug/kg
CIS-1,3-DICHLOROPROPENE	BDL	25	ug/kg
DICHLORODIFLUOROMETHANE	BDL	25	ug/kg
1,1-DICHLOROETHANE	BDL	25	ug/kg
1,2-DICHLOROETHANE	BDL	25	ug/kg
1,1-DICHLOROETHENE	BDL	25	ug/kg
1,2-DICHLOROPROPANE	BDL	25	ug/kg
ETHYLBENZENE	BDL	25	ug/kg
FLUOROTRICHLOROMETHANE	BDL	25	ug/kg
2-HEXANONE	BDL	50	ug/kg
METHYLENE CHLORIDE	42	25	ug/kg
METHYL ETHYL KETONE	BDL	50	ug/kg
4-METHYL-2-PENTANONE	BDL	50	ug/kg
STYRENE	BDL	25	ug/kg
1,1,2,2-TETRACHLOROETHANE	BDL	25	ug/kg
TETRACHLOROETHENE	320	25	ug/kg
TETRAHYDROFURAN	BDL	120	ug/kg
TOLUENE	75	25	ug/kg
1,2-DICHLOROETHENE (TOTAL)	BDL	25	ug/kg
TRANS-1,3-DICHLOROPROPENE	BDL	25	ug/kg
1,1,1-TRICHLOROETHANE	710	25	ug/kg
1,1,2-TRICHLOROETHANE	BDL	25	ug/kg

HERITAGE LABORATORIES, INC.

Lab Sample ID: A293253

Parameter	Result	Det. Limit	Units
TRICHLOROETHENE	73	25	ug/kg
VINYL ACETATE	BDL	50	ug/kg
VINYL CHLORIDE	BDL	50	ug/kg
XYLENE (TOTAL)	BDL	25	ug/kg
.			
SURROGATE RECOVERY			

DICHLOROETHANE-D4	95		% Rec
TOLUENE-D8	104		% Rec
BROMOFLUOROBENZENE	98		% Rec

1:5 DILUTION.

Sample Comments

BDL Below Detection Limit

Sample chain of custody number 10936.

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CHARLES JACKSON, QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555, ROCKVILLE, IN 47872

Quality Assurance Officer: _____

HERITAGE LABORATORIES, INC.

Lab Sample ID: A293253

LIST OF COMPLETED TASKS

GC/MS CLP GC/MS CLP

Completed 03-NOV-93

Last Task Page

CERTIFICATE OF ANALYSIS

Service Location

HERITAGE LABORATORIES, INC.
7901 W. MORRIS ST.
INDIANAPOLIS, IN 46231
(317)243-8305

Received	Project	Lab ID
28-OCT-93	2506	A294793
Complete	PO Number	
08-NOV-93		
Printed	Sampled	
09-NOV-93	19-OCT-93 16:30	

Report To

ROBERT J. AUTIO
QUALITY ENVIRONMENTAL MANAGEMENT
1640 STRICKLAND
MARTINSVILLE, IN 46151

Bill To

CHARLES JACKSON
QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555
ROCKVILLE, IN 47872

Sample Description

DESCRIPTION: HA-3 (0.5-1.0')
LOCATION: ENVIROCHEM - SITE PREP & MATERIAL REMOVAL

VOLATILE ORGANICS (HEATED PURGE & TRAP) SW846-8240A

Analyst: G. WILSON Analysis Date: 01-NOV-93 11:05 Instrument: GC/MS VOA Test: 0510.9.0

Parameter	Result	Det. Limit	Units
ACETONE	BDL	20	ug/kg
ACROLEIN	BDL	50	ug/kg
ACRYLONITRILE	BDL	70	ug/kg
BENZENE	BDL	5	ug/kg
BROMODICHLOROMETHANE	BDL	5	ug/kg
BROMOFORM	BDL	5	ug/kg
BROMOMETHANE	BDL	10	ug/kg
CARBON DISULFIDE	BDL	5	ug/kg
CARBON TETRACHLORIDE	BDL	5	ug/kg
CHLOROBENZENE	BDL	5	ug/kg
CHLOROETHANE	BDL	10	ug/kg
CHLOROFORM	BDL	5	ug/kg
CHLOROMETHANE	BDL	10	ug/kg
DIBROMOCHLOROMETHANE	BDL	5	ug/kg
CIS-1,3-DICHLOROPROPENE	BDL	5	ug/kg
DICHLORODIFLUOROMETHANE	BDL	5	ug/kg
1,1-DICHLOROETHANE	BDL	5	ug/kg
1,2-DICHLOROETHANE	BDL	5	ug/kg
1,1-DICHLOROETHENE	BDL	5	ug/kg
1,2-DICHLOROPROPANE	BDL	5	ug/kg
ETHYLBENZENE	BDL	5	ug/kg
FLUOROTRICHLOROMETHANE	BDL	5	ug/kg
2-HEXANONE	BDL	10	ug/kg
METHYLENE CHLORIDE	8	5	ug/kg
METHYL ETHYL KETONE	BDL	10	ug/kg
4-METHYL-2-PENTANONE	BDL	10	ug/kg
STYRENE	BDL	5	ug/kg
1,1,2,2-TETRACHLOROETHANE	BDL	5	ug/kg
TETRACHLOROETHENE	BDL	5	ug/kg
TETRAHYDROFURAN	BDL	25	ug/kg
TOLUENE	20	5	ug/kg
1,2-DICHLOROETHENE (TOTAL)	BDL	5	ug/kg
TRANS-1,3-DICHLOROPROPENE	BDL	5	ug/kg
1,1,1-TRICHLOROETHANE	74	5	ug/kg

Parameter	Result	Det. Limit	Units
1,1,2-TRICHLOROETHANE	BDL	5	ug/kg
TRICHLOROETHENE	21	5	ug/kg
VINYL ACETATE	BDL	10	ug/kg
VINYL CHLORIDE	BDL	10	ug/kg
XYLENE (TOTAL)	BDL	5	ug/kg
SURROGATE RECOVERY			
DICHLOROETHANE-D4	111		% Rec
TOLUENE-D8	98		% Rec
BROMOFLUOROBENZENE	103		% Rec

Sample reanalyzed with no improvement in internal standard areas.

Sample Comments

BDL Below Detection Limit

Sample chain of custody number 13243.

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CHARLES JACKSON, QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555, ROCKVILLE, IN 47872

Quality Assurance Officer: GABusch

CERTIFICATE OF ANALYSIS

Service Location

HERITAGE LABORATORIES, INC.
7901 W. MORRIS ST.
INDIANAPOLIS, IN 46231
(317)243-8305

Received	Project	Lab ID
29-OCT-93	2506	A295096
Complete	**PO Number**	
11-NOV-93	9311001-RJA	
Printed	**Sampled**	
11-NOV-93	29-OCT-93 14:25	

Report To

ROBERT J. AUTIO
QUALITY ENVIRONMENTAL MANAGEMENT
1640 STRICKLAND
MARTINSVILLE, IN 46151

Bill To

CHARLES JACKSON
QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555
ROCKVILLE, IN 47872

Sample Description

DESCRIPTION: HA-16 (1.5-2.0')

VOLATILE ORGANICS (HEATED PURGE & TRAP) SW846-8240A

Analyst: G. WILSON Analysis Date: 04-NOV-93 06:10 Instrument: GC/MS VOA Test: 0510.9.0

Parameter	Result	Det. Limit	Units
ACETONE	BDL	20	ug/kg
ACROLEIN	BDL	50	ug/kg
ACRYLONITRILE	BDL	70	ug/kg
BENZENE	BDL	5	ug/kg
BROMODICHLOROMETHANE	BDL	5	ug/kg
BROMOFORM	BDL	5	ug/kg
BROMOMETHANE	BDL	10	ug/kg
CARBON DISULFIDE	BDL	5	ug/kg
CARBON TETRACHLORIDE	BDL	5	ug/kg
CHLOROBENZENE	BDL	5	ug/kg
CHLOROETHANE	BDL	10	ug/kg
CHLOROFORM	BDL	5	ug/kg
CHLOROMETHANE	BDL	10	ug/kg
DIBROMOCHLOROMETHANE	BDL	5	ug/kg
CIS-1,3-DICHLOROPROPENE	BDL	5	ug/kg
DICHLORODIFLUOROMETHANE	BDL	5	ug/kg
1,1-DICHLOROETHANE	BDL	5	ug/kg
1,2-DICHLOROETHANE	BDL	5	ug/kg
1,1-DICHLOROETHENE	BDL	5	ug/kg
1,2-DICHLOROPROPANE	BDL	5	ug/kg
ETHYLBENZENE	BDL	5	ug/kg
FLUOROTRICHLOROMETHANE	BDL	5	ug/kg
2-HEXANONE	BDL	10	ug/kg
METHYLENE CHLORIDE	5	5	ug/kg
METHYL ETHYL KETONE	BDL	10	ug/kg
4-METHYL-2-PENTANONE	BDL	10	ug/kg
STYRENE	BDL	5	ug/kg
1,1,2,2-TETRACHLOROETHANE	BDL	5	ug/kg
TETRACHLOROETHENE	BDL	5	ug/kg
TETRAHYDROFURAN	BDL	25	ug/kg
TOLUENE	6	5	ug/kg
1,2-DICHLOROETHENE (TOTAL)	BDL	5	ug/kg
TRANS-1,3-DICHLOROPROPENE	BDL	5	ug/kg
1,1,1-TRICHLOROETHANE	11	5	ug/kg
1,1,2-TRICHLOROETHANE	BDL	5	ug/kg

HERITAGE LABORATORIES, INC.

Lab Sample ID: A295096

Parameter	Result	Det. Limit	Units
TRICHLOROETHENE	7	5	ug/kg
VINYL ACETATE	BDL	10	ug/kg
VINYL CHLORIDE	BDL	10	ug/kg
XYLENE (TOTAL)	BDL	5	ug/kg
SURROGATE RECOVERY			
DICHLOROETHANE-D4	94		% Rec
TOLUENE-D8	103		% Rec
BROMOFLUOROBENZENE	92		% Rec

Sample Comments

BDL Below Detection Limit

Sample chain of custody number 14434.

This Certificate shall not be reproduced, except in full, without the written approval of the lab.

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CHARLES JACKSON, QUALITY ENVIRONMENTAL MANAGEMENT
RR 1, BOX 555, ROCKVILLE, IN 47872

Quality Assurance Officer: _____

HERITAGE LABORATORIES, INC.

Lab Sample ID: A295096

LIST OF COMPLETED TASKS

GC/MS CLP	GC/MS CLP	Completed 11-NOV-93

APPENDIX B

CEIMIC CORPORATION
SOP NUMBER 004

GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS

CEIMIC CORPORATION

TITLE: GAS CHROMATOGRAPHY/MASS SPECTROMETRY FOR VOLATILE ORGANICS

REFERENCES:

1. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, OLM01.8

1.0 SCOPE AND APPLICATIONS

1.1 This method is used to determine volatile organic compounds (VOC's) in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, solids, and sediments.

1.1.1 Analysis of water and soil samples must be completed within ten (10) days of sample receipt.

1.2 This method can be used to quantify most VOC's that have boiling points below 200°C (vapor pressure is approximately equal to mm Hg @ 25°C) and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for the more soluble compounds, quantitation limits are approximately 10 times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a gas chromatograph (GC) column packed with graphitized carbon lightly coated with a carbowax. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Table 4-1 for a list of compounds, retention times, and their characteristic ions that have been evaluated on a purge-and-trap gas chromatograph/mass spectrometer (GC/MS) system.

1.3 The quantitation limits of this method are 10 ug/Kg for low soil, 10 ug/L for low water, and 1200 ug/Kg for medium soil.

1.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and GC/MS's and who are skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.5 To increase purging efficiencies of acrylonitrile and acrolein, a heated purge step is recommended.

2.0 SUMMARY OF METHOD

2.1 Water samples

2.1.1 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a GC column. The GC is temperature-programmed to separate the purgeables, which are then detected with an MS.

2.1.2 An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 ml aliquot of the dilution is taken for purging.

2.1.3 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a specially designed purging chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.2 Soil/Sediment Samples

2.2.1 Low level

An inert gas is bubbled through a mixture of a 5 g sample and 5 ml reagent water contained in a heated soil purging chamber at elevated temperatures. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a GC column. The GC is temperature-programmed to separate the purgeables, which are then detected with an MS.

2.2.2 Medium Level

A 4 g measured amount of soil is extracted with 9 ml methanol and 1 ml surrogate standard. A portion (100 ul) of the methanol extract is diluted to 5 ml with reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber at ambient temperature. The purgeables are effectively transferred from the aqueous phase to the vapor phase. The vapor

is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a GC column. The GC is temperature-programmed to separate the purgeables, which are then detected with an MS.

3.0 INTERFERENCES AND POTENTIAL PROBLEMS

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check on such contamination. One holding blank per case is analyzed. The data is retained by the laboratory and is available for inspection at the client's request.

3.3 Contamination by carryover can occur whenever high- and low-level samples are sequentially analyzed. To reduce carryover, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross-contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents.

3.5 Manual integration may be necessary when peaks are missed by the automatic quantitation algorithms. Instances of manual integration are documented in the case narrative, and copies of the manual integration are dated, initialled and placed in the case file.

3.5.1 Manual integration is performed by first locating the appropriate peak.

3.5.2 The GC/MS Operator integrates, to the best of their ability, the area of the extracted ion current profile of the quanitation ion characteristic to that analyte. This is accomplished with no regard to acceptance criteria.

3.5.3 All manual integrations are flagged with an "M" on the Quantitation Report.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes: 10 ul, 25 ul, 50 ul, 100 ul, 250 ul, 500 ul, and 1,000 ul. These syringes should be equipped with a 20-gauge (0.006-inch I.D.) needle.

4.2 Syringe valve: Two-way, with Luerlok ends (3 each), if applicable to the purging device

4.3 Syringe: 5-ml glass gastight

4.4 Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

4.5 Glass Scintillation Vials: 20 ml, with screw caps and Teflon liners or glass culture tubes with a screw cap and Teflon liner

4.6 Volumetric Flasks: 10 ml, 25 ml, and 100 ml, Class A with ground-glass stoppers

4.7 Vials: 40 ml, with pierceable Teflon screw cap top

4.8 Spatula: Stainless steel

4.9 Disposable Pipettes: Pasteur

4.10 Chamber Heater: Tekmar sampler heater, capable of maintaining the purging chamber to within 1°C over the temperature range of ambient to 100°C

4.11 pH Paper: Wide Range

4.12 Purge and Trap Device: Tekmar LSC2000/ALS 2016

4.12.1 The sample purger is designed to accept 5 ml samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap has a total volume of less than 15 ml. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas is introduced no more than 5 mm from the base of the water column.

4.12.2 The trap is at least 25 cm long and has an inside diameter of at least 0.105 inches. The trap is packed to contain the following minimum lengths of absorbents: 15 cm Tenax-GC (60/80 mesh) and 8 cm Silica Gel molecular sieve (35/60 mesh).

4.12.3 The desorber is capable of rapidly heating the trap to 220°C and performing the bakeout step at 260°C.

4.12.4 The chamber heater is capable of maintaining the purge device at 40°C ± 1°C.

4.13 GC (Hewlett Packard 5890)

A complete analytical system with a temperature programmable GC suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

4.14 Column

4.14.1 6 feet long x 0.1 inches ID glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh)

4.14.2 30 m X 0.543 mm ID, DB624, commercially available from J&W Scientific

4.15 Mass Spectrometer (Hewlett Packard 5970MSD)

Capable of scanning from 35 to 300 amu every 2 seconds or less for capillary column and every 3 seconds or less for packed columns, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria listed below when 50 ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet.

BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	8.0% - 40% of mass 95
75	30% - 66% of mass 95
95	Base peak, 100% relative abundance
96	5.0% - 9.0% of mass 95
173	Less than 2.0% of mass 174
174	50.0% - 120.0% of mass 95
175	4.0% - 9.0% of mass 174
176	93.0% - 101.0% of mass 174
177	5.0% - 9.0% of mass 176

4.16 GC/MS Interface

Constructed of glass-lined material which has been deactivated by silanizing with dichlorodimethylsilane.

4.17 Data System (Hewlett Packard RTE-A100 Series)

This system is interfaced to the MS and allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer's software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. A Hewlett Packard magnetic tape storage device is used to archive data.

5.0 REAGENTS

5.1 Stock Solutions

Stock solutions are prepared from pure standard materials or purchased as certified solutions (See SOP009). Stock standard solutions are prepared in methanol, using assayed liquids.

5.1.1 Using a 100 ul syringe, immediately add 2 or more drops of assayed reference material into a 10 ml volumetric flask filled to the neck with methanol; reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask. The amount transferred should be 250 $\pm$ 10 mg.

5.1.2 Reweigh, dilute to volume, stopper, and mix by inverting the flask 4 times. Calculate the concentration in ug/ul from the net gain in weight. When compound purity is assayed to be

≥97%, the weight may be used without correction to calculate the concentration of the stock standard. For gaseous compounds, calculate the concentration in micrograms per microliter, using the Ideal Gas Law, taking into account the temperature and pressure conditions within the laboratory.

5.1.3 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.1.4 Fresh standards are prepared every 2 months for gases. Reactive compounds such as 2-chloroethylvinyl ether and styrene are prepared more frequently. All other standards are replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.1.5 The following standards are purchased neat to be assayed >97% and prepared per Sections 5.1.1 to 5.1.4.

5.1.5.1 Surrogate Standards (Section 5.3)

5.1.5.2 Internal Standards (Section 5.4)

5.1.5.3 BFB Standard (Section 5.5)

5.1.5.4 Matrix Spiking Standard (Section 5.7)

5.1.5.4.1 1,1-Dichloroethene

5.1.5.4.2 Trichloroethene

5.1.5.4.3 Chlorobenzene

5.1.5.4.4 Toluene

5.1.5.4.5 Benzene

5.1.6 The following calibration standards are obtained from a commercial source. These are stored in sealed ampules at -10°C to -20°C in the freezer located in the VOA Laboratory. These standards are used to prepare the initial and continuing calibration standards; no secondary dilution standard is prepared from these except for the HSL standard (Section 5.2).

5.1.6.1 Purgeable A, Supelco, Cat. #4-8851M, 200 ug/ml

Carbon tetrachloride	1,2-Dichloropropane
Chlorobenzene	Methylene chloride
2-Chloroethylvinyl ether	Tetrachlorethylene
Chloroform	1,1,2-Trichloroethane
Dibromochloromethane	Trichloroethylene
1,1-Dichloroethane	Trichlorofluoromethane
1,1-Dichloroethylene	

5.1.6.2 Purgeable B, Supelco, Cat. #4-8852M, 200 ug/ml

Benzene	Cis-1,3-dichloropropene
Bromodichloromethane	Ethyl benzene
Bromoform	1,1,2,2-Tetrachloroethane
1,2-Dichloroethane	Toluene
Trans-1,2-dichloroethylene	1,1,1-Trichloroethane

5.1.6.3 Purgeable C, Supelco, Cat. #4-8853M, 200 ug/ml

Bromomethane	Chloromethane
Chloroethane	Vinyl chloride

5.1.6.4 TCL Volatile Mix #1, Supelco, Cat. #48949, 2,000 mg/ml

Acetone	2-Butanone
4-Methyl-2-pentanone	2-Hexanone

5.1.6.5 M-Xylene, Supelco, Cat. #4-0202M, 5,000 ug/ml

5.1.6.6 P-Xylene, Supelco, Cat. #4-0203M, 5,000 ug/ml

5.1.6.7 O-Xylene, Supelco, Cat. #4-0201M, 5,000 ug/ml

5.1.6.8 Carbon Disulfide, Supelco, Cat. #4-0363M, 5,000 ug/ml

5.1.6.9 Styrene, Supelco, Cat. #4-0257M, 5,000 ug/ml

5.1.6.10 cis-1,2-dichloroethene, Supelco, Cat. #4-0173

5.2 Secondary Dilution Standards

Using stock standard solutions from Sections 5.1.6.4, 5.1.6.6, and 5.1.6.7 or 5.1.6.5, 5.1.6.6, 5.1.6.7, 5.1.6.8, 5.1.6.9, 5.1.6.10, and 5.1.6.11, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. Secondary dilution standards are stored with minimal headspace and checked frequently for signs of degradation or evaporation. Fresh secondary dilution standards for gases and reactive

compounds should be prepared every month or sooner, if the standard has degraded or evaporated.

5.2.1 HSL Standard

5.2.1.1 Alternately from TCL Volatile Mix #1 (5.1.6.5), transfer 1,000 ul into a clean 10 ml. volumetric flask containing 5 ml of methanol. Add 400 ul of m-xylene (Section 5.1.6.6), 400 ul of p-xylene (Section 5.1.6.7), 400 ul of o-xylene (Section 5.1.6.8), 400 ul of carbon disulfide (Section 5.1.6.9), and 400 ul of styrene (Section 5.1.6.10) and 400 ul of cis-1,2-dichloroethene (Section 5.1.6.10) and make up to the 10 ml mark with methanol.

5.2.2 Surrogate, Internal, and BFB Standard Preparation.

5.2.2.1 Stock standards are prepared separately per Sections 5.1.1 to 5.1.4 to give a concentration of 25,000 ug/ml.

5.2.2.2 An aliquot of this solution is diluted in methanol per Section 5.2 (Refer to specific Sections 5.3, 5.4, 5.5, and 5.7).

5.2.3 Matrix Spike Solutions

5.2.3.1 Stock standards are prepared separately per Sections 5.1.1 to 5.1.4 to give a concentration of 25,000 ug/ml.

5.2.3.2 An aliquot of these solutions are diluted in methanol per Section 5.7.

5.3 System Monitoring Compound Standards

5.3.1 The system monitoring compounds are toluene-d_8, BFB, and 1,2-dichloroethane-d_4.

5.3.2 Individual stock system monitoring compound solutions in methanol are prepared as described in Section 5.1.

5.3.3 Several system monitoring compound standard solutions are prepared from the stock at different concentrations in methanol per Section 5.2.2 and below.

5.3.3.1 Medium Soils

Add 10 ul from each stock system monitoring compound solution prepared in Sections 5.1.1

to 5.1.4 to a 10 ml volumetric flask containing 9 ml of methanol, and make up to the mark with methanol to give a concentration of 25 ug/ml. One ml of this standard is added to 9 ml of methanol to perform the extraction.

5.3.3.2 System Monitoring Compound Calibration Standard

Add 80 ul of each stock system monitoring compound solution prepared in Sections 5.1.1 to 5.1.4 to a 10 ml volumetric flask containing 9 ml of methanol and make up to the mark with methanol. This is a concentration of 200 ug/ml and is used for 5-point calibrations only.

5.3.3.3 System Monitoring Compound/Internal Standard QC Mix

This solution is prepared by adding 10 ul from each stock system monitoring compound and each internal standard solution to a 10 ml volumetric flask containing 9 ml of methanol and making up to the mark with methanol. This QC mix contains 25 ug/ml of each system monitoring compound and each internal standard (refer to Section 5.4.3.2). 10 ul of this solution spiked into 5 ml of sample yields a concentration of 50 ppb of each system monitoring compound and each internal standard.

5.4 Internal Standards

5.4.1 The internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5.

5.4.2 Individual stock internal standard solutions in methanol are prepared as described in Section 5.1.

5.4.3 Two internal standard solutions are prepared from the stock standard methanol per Section 5.2.2 and below.

5.4.3.1 Internal Standard Solution for Calibration

Add 10 ul from the stock solution of each internal prepared in Sections 5.1.1 to 5.1.4 to a 10 ml volumetric flask containing 9 ml of methanol and make up to the mark with methanol to give a concentration of 25 ug/ml of each internal standard compound. Addition

of 10 ul of this standard to 5 ml of calibration standard would be the equivalent of 50 ppb. This internal standard solution is used for 5-point calibrations and medium-level soils. This spiking standard must be prepared weekly or sooner if the solution has degraded or evaporated.

5.4.3.2 System Monitoring Compound/Internal Standard QC Mix

See Section 5.3.3.3

5.5 BFB Standard

5.5.1 A stock solution in methanol is prepared as described in Section 5.1 to give a concentration of 25,000 ug/ml.

5.5.2 Add 10 ul from the stock solution prepared in Sections 5.1.1 to 5.1.4 to a 10 ml volumetric flask containing 9 ml of methanol and make up to the mark with methanol to give a concentration of 25 ug/ml. Inject 2 ul, a total amount of 50 ng of BFB, for the tune.

5.6 Calibration Standards

Calibration standards at 5 concentration levels in water are prepared from the stock solutions A,B, and C and secondary dilution standard HSL (Section 5.2.1) and the surrogate calibration standard (Section 5.3.3.2). Stock solutions are stabilized at room temperature. The 10 ul of the 10 ml internal standard solution prepared in Section 5.4.3.1 is added prior to the purge step.

Analyte Concentration*	Final Aqueous Volume	Added Volume of A,B,C,HSL, Surrogate	Syringes
10 ppb	100 ml	5 ul	10 ul
20 ppb	100 ml	10 ul	25 ul
50 ppb	100 ml	25 ul	50 ul
100 ppb	100 ml	50 ul	50 ul
200 ppb	25 ml	25 ul	50 ul

* The appropriate volume of each stock solution above and HSL secondary dilution standard is added to a 100 ml or 25 ml flask containing an aliquot of organic free water. The flask is then made up to the mark, inverted 3 times, and the neck volume of the volumetric is discarded prior to loading the 5 ml sample syringe.

5.7 Matrix Spiking Standard

5.7.1 The following compounds are used as matrix spike compounds: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

5.7.2 Individual stock matrix spike compound solutions in methanol are prepared as described in Section 5.1.

5.7.3 The working matrix spike solution is prepared from the individual stock standards in methanol, per Section 5.2.3. Add 10 ul of each stock matrix spike compound solution to a 10 ml volumetric flask containing 9 ml of methanol, and made up to the mark with methanol to give a concentration of 25 ug/ml of each matrix spike compound. Addition of 10 ul of this solution spiked into 5 ml sample yields a concentration of each analyte at 50 ppb.

5.8 Great care is taken to maintain the integrity of all standard solutions. All standards are stored at -10°C to -20°C in screw-cap amber bottles with Teflon liners, with zero headspace.

5.9 Organic-Free Water

Reagent water is defined as water in which an interferant is not observed at the method detection limit (MDL) of the parameters of interest. Tap water, filtered through activated charcoal meets the above criteria.

5.10 Methanol

Methanol for Purge and Trap Analysis (Burdick and Jackson) which is better than Pesticide quality. Store apart from other solvents.

5.11 Storage of Standards

5.11.1 Store the stock standards in Teflon-sealed screw-cap bottles with zero headspace at -10°C to -20°C. Protect the standards from light. Once one of the bottles containing the stock standard solution has been opened, it may be used for no longer than one week.

5.11.2 Store secondary dilution standards in Teflon-sealed screw cap bottles with minimal headspace at -10°C to -20°C. Protect the

standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing the working calibration standards from them.

5.11.3 Aqueous standards may be stored for up to 24 hours if held in Teflon-sealed screw-cap vials with zero headspace at 4°C. Protect the standards from light. If not so stored, they must be discarded after one hour unless they are set up to be purged by an autosampler. When using an autosampler, the standards may be kept for up to 12 hours in purge tubes connected via the autosampler to the purge and trap device.

5.11.4 Purgeable standards must be stored separately from other standards.

6.0 PROCEDURE

6.1 Direct Injection

In very limited applications (e.g., aqueous process wastes), direct injection of the sample into the GC/MS system with a 10 ul syringe may be appropriate. One such application is for verification of the alcohol content of an aqueous sample prior to determining if the sample is ignitable (Methods 1010 or 1020). In this case, it is suggested that direct injection be used. The detection limit is very high (approximately 10,000 ug/l); therefore, it is only permitted when concentrations in excess of 10,000 ug/l are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

6.2 Initial Calibration for Purge-and-Trap Procedures

6.2.1 Recommended GC/MS Operating Conditions

Electron energy	70 volts (nominal)
Mass range	35-300 amu
Scan time	2.5 sec./scan (1.5 sec./scan for capillary)
Initial column temperature	45°C (10° for capillary)

Column Hold time	Until last compound elutes
Initial column holding time	3 minutes
Column temperature program	8°C/min. (6°C/min. for capillary)
Final column temperature	220°C (160°C for capillary)
Final column holding time	15 minutes (26 min. for capillary)
Injector temperature	150°C (125°C for capillary)
Transfer line temperature	250°C
Carrier gas	Helium at 30 cm^3/min. (15 cm^3/min for capillary)

6.2.2 Each GC/MS system must be hardware-tuned to meet the criteria in Section 4.15 for a 50 ng injection of BFB (2 ul injection of the BFB standard). Once every 12 hours, the mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Analyses must not begin until these criteria are met.

6.2.3 Assemble a purge-and-trap device that meets the specification in Section 4.12. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 ml/minute. Prior to use, condition the trap daily for 10 minutes while backflushing at 180°C with the column at 220°C (160°C for capillary).

6.2.4 Connect the purge-and-trap device to a GC.

6.2.5 Prepare the final solutions containing the required concentrations of calibration

standards (Section 5.6), including system monitoring compounds standards, according to Section 5.3.3.2 system monitoring compounds calibration standard is used for the initial calibration so that the concentration of system monitoring compounds is the same as the concentration of the target compounds.

Fill a 5 ml glass syringe with the individual standard and remove air and excess water. Add 10 ul of the internal standard solution (Section 5.4.3.1) with a 10 ul syringe to the 5 ml of standard and transfer to the purge-and-trap chamber. A water chamber is used for aqueous and medium soils, a heated soil chamber for low soils.

6.2.6 Carry out the purge-and-trap analysis procedure as described in Section 6.4.1.

6.2.7 Tabulate the area response of the characteristic ions (see Table 4-1) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to its internal standards (See Table 1).

The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

Where:

RF = relative response factor

A_x = area of the characteristic ion for the compound being measured

A_{is} = areas of the characteristic ion for the specific internal standard

C_{is} = concentration of the specific internal standard

C_x = concentration of the compound being measured

Calculating the relative response factor of the Xylenes and the cis and trans isomers of 1,2-Dichloroethene requires special attention. On packed columns, o-and p-Xylene isomers coelute. On capillary columns, the m and p isomers coeulte. Therefore, when calculating the relative response factor in

the equation below, use the area response (A_x) and concentration (C_x) of the peak that represents the single isomer on the GC column used for analysis.

For the cis and trans isomers of 1,2-Dichloroethene which may coelute on packed columns but not on capillary columns, both isomers must be present in the standards. If the two isomers coelute, use the area of the coeluting peak and the total concentration of the two isomers in the standard to determine the relative response factor. If the two isomers do not coelute, sum the areas of the two peaks and the concentrations of the two isomers in the standard to determine the relative response factor.

TABLE 1
Compounds With Their Associated IS

BROMOCHLOROMETHANE	1,4-DIFLUOROBENZENE	CHLOROBENZENE-D_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-pentanone
Vinyl Chloride		Tetrachloroethene
Chloroethane	Bromodichloromethane	1,1,2,2-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	Trans-1,3-dichloropropene	Chlorobenzene
Carbon disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Xylene (total)
1,2-Dichloroethene (Tot)	Benzene	BFB (Surr)
Chloroform	Cis-1,3-dichloropropene	Toluene-d_8 (Surr)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (Surr)	Bromoform	

(Surr) = Surrogate compound

6.2.8 Using the RF's from the initial calibration, calculate the percent relative standard deviation (% RSD) for each compound.

$$\% \text{ RSD} = \frac{SD}{X} \times 100$$

Where:

RSD = relative standard deviation

X = mean of 5 initial RF's for a compound

SD = standard deviation of average RF's for a compound

6.2.9 Relative response criteria for initial and continuing calibration are listed as follows in Table 2.

TABLE 2
RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF VOLATILE ORGANIC COMPOUNDS

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Bromomethane	0.100	20.5	25.0
Vinyl chloride	0.100	20.5	25.0
1,1-Dichloroethene	0.100	20.5	25.0
1,1-Dichloroethane	0.200	20.5	25.0
Chloroform	0.200	20.5	25.0
1,2-Dichloroethane	0.100	20.5	25.0
1,1,1-Trichloroethane	0.100	20.5	25.0
Carbon Tetrachloride	0.100	20.5	25.0
Bromodichloromethane	0.200	20.5	25.0
cis-1,3-Dichloropropene	0.200	20.5	25.0
Trichloroethene	0.300	20.5	25.0
Dibromochloromethane	0.100	20.5	25.0
1,1,2-trichloroethane	0.100	20.5	25.0
Benzene	0.500	20.5	25.0
trans-1,3-Dichloropropene	0.100	20.5	25.0
Bromoform	0.100	20.5	25.0
Tetrachloroethene	0.200	20.5	25.0
1,1,2,2-Tetrachloroethane	0.500	20.5	25.0
Toluene	0.400	20.5	25.0
Chlorobenzene	0.500	20.5	25.0
Ethylbenzene	0.100	20.5	25.0
Styrene	0.300	20.5	25.0
Xylenes (total)	0.300	20.5	25.0
Bromofluorobenzene	0.200	20.5	25.0

6.2.10 The following compounds have no Maximum % RSD, or Maximum %Difference criteria; however, these compounds must meet a minimum RRF criterion of 0.010:

Acetone
2-Butanone
Carbon disulfide
Chloromethane
1,2-Dichloroethene(total)
1,2-Dichloropropane
Methylene chloride
2-Hexanone
Toluene-d_8
Chloroethane
4-Methyl-2-pentanone
1,2-Dichloroethaned4

6.3 Daily GC/MS Calibration

6.3.1 Prior to the analysis of samples, inject 50 ng of the BFB standard. The resultant mass spectra for the BFB must meet all of the criteria given in Section 4.15 before sample analysis begins. These criteria must be demonstrated each 12-hour shift.

6.3.2 The initial calibration curve (Section 6.2) for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a continuing calibration standard at a concentration of 50 ug/l and by checking to see that the criteria of 6.2.10 and 6.2.11 are met. If the criteria are not met, the system must be evaluated and corrective action must be initiated.

6.3.3 Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

6.3.4 The internal standard responses and retention times in the continuing calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last continuing calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of 2 (-50% to +100%) from the last continuing calibration standard, the MS must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

6.3.5 Method blanks analysis must be performed as follows:

6.3.5.1 For water samples, a volatile method blank consists of a 5 mL volume of reagent water spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

6.3.5.2 For low level soil/sediment samples, a volatile method blank consists of a 5 gm of a purified solid matrix added to reagent water, spiked with the system monitoring compounds and internal standards, and carried through the analytical procedure.

6.3.5.3 For medium level soil/sediment samples, a volatile method blank consists of 4 gm of a purified solid matrix spiked with the system monitoring compounds, extracted with methanol, and carried through the analytical procedure.

6.3.5.4 An acceptable volatile method blank for samples must contain less than or equal to five times (5x) the Contract Required Quantitation Limit (CRQL) of Methylene Chloride, Acetone, and 2-Butanone, and less than or equal to the CRQL of any other volatile target compound.

6.3.5.5 For the analysis of volatile compounds, a method blank analysis must be performed once for each 12-hour time period, immediately after the continuing calibration to assure there is no carryover into samples.

6.3.5.6 If a laboratory method blank exceeds the above criteria, the operator must consider the analytical system to be out of control. The source of the contamination must be investigated, and appropriate corrective measures MUST be taken and documented before further sample analysis. All samples processed with a method blank that is out of control (i.e., contaminated) MUST be reextracted/repurged and reanalyzed.

6.4 GC/MS Analysis

6.4.1 Water samples

6.4.1.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

6.4.1.2 Set up the GC/MS system as outlined in Section 6.2.1.

6.4.1.3 BFB tuning criteria and daily GC/MS calibration criteria must be met (Section 6.3) before analyzing samples.

6.4.1.4 Adjust the purge gas (helium) flow rate to 25-40 ml/minute on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response (see Section 6.2.8).

6.4.1.5 Remove the plunger from a 5 ml syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing, replace the plunger, and remove air and excess water.

6.4.1.6 Add 10 ul of surrogate/internal standard QC mix (Section 5.3.3.3). The addition of 10 ul of the surrogate/internal standard QC mix to 5 ml of sample is equivalent to a concentration of 50 ug/l of each surrogate and internal standard.

6.4.1.7 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber.

6.4.1.8 Close both valves and purge the sample for 11 ($\pm$ 0.1) minutes at ambient temperature.

6.4.1.9 Check the pH of the sample after the aliquot has been taken for the analysis. The pH is determined by placing a few drops of sample onto pH paper. Document the results in the pH Analysis Logbook (Figure 4-2) and include in the SDG Narrative.

6.4.1.10 At the conclusion of the purge time, the purge-and-trap will signal the GC and begin to desorb the trap while initiating the chromatographic temperature program and the MS data acquisition.

6.4.1.11 While the trap is being desorbed, or later, wash the chamber with a minimum of 3 5-ml flushes of reagent water.

6.4.1.12 After the sample has been desorbed for 4 minutes, the purge-and-trap will recondition the trap by heating to 180°C while backflushing. This step will take 7 minutes.

6.4.1.13 If a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. The following procedure is followed for diluting volatile organic samples.

6.4.1.13.1 Dilutions must be made in volumetric flasks (10 to 100 ml). Select the volumetric flask that will allow for the necessary dilution. Step dilutions may be necessary for extremely large dilutions.

6.4.1.13.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected, and add slightly less than this quantity of reagent water to the flask.

6.4.1.13.3 Inject the proper aliquot of sample from the sample vial into the flask. Aliquots of less than 1 ml are not recommended. Dilute the sample to the mark with reagent water. Cap the flask and invert 3 times. Repeat above procedure for additional dilutions.

6.4.1.13.4 Fill a 5 ml syringe with the diluted sample as in Section 6.4.1.6.

6.4.1.14 When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by an organic-free water analysis. If this blank analysis is not free of interferences, the system must be decontaminated.

Sample analysis may not resume until a blank can be analyzed that is free of interferences.

6.4.1.15 For matrix spike analysis, add 10 ul of the matrix spike solution (Section 5.7) to the 5 ml of sample and 10 ul of QC mixture and purge. Disregarding any dilution, this is equivalent to a concentration of 50 ug/l of each matrix spike compound.

6.4.1.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Sections 6.5.1 and 6.5.2 for qualitative and quantitative analysis.

6.4.2 Water-miscible Liquids

6.4.2.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

6.4.2.2 Initial and serial dilutions can be prepared by pipetting 2 ml of the sample to a 100 ml volumetric flask and diluting to volume with reagent water. Transfer immediately to a 5 ml syringe.

6.4.2.3 Alternatively, prepare dilutions directly in a 5 ml syringe filled with reagent water by adding at least 20 ul, but not more than 100 ul, of liquid sample. The sample is ready for addition of the surrogate/internal standard QC mix.

6.4.3 Sediment/Soil and Waste Samples

Weigh 5.0 gm of soil into a soil analysis chamber. If peaks are saturated from the analysis of a 5 g sample, a smaller sample size must be analyzed to prevent saturation. However, the smallest sample size permitted is 1 g. Record this in the Soil Preparation Logbook.

6.4.3.1. Low-Level Method

6.4.3.1.1 The GC/MS system should be set up as in Sections 6.4.1.1 - 6.4.1.4. This should be done prior to the

preparation of the sample to avoid loss of volatiles from standards and sample. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions (Section 5.3), but use a 40°C purge temperature. Note: Aqueous samples and medium level soil samples do not use heated purge.

6.4.3.1.2 Organic-free water containing surrogates and internal standards is added to the soil. A 5 ml syringe is filled to just short of overflowing with organic-free water and air and excess water are removed. Add 10 ul each of the surrogate/internal standard QC mix (Section 5.3.3.3) to the syringe. The addition of 10 ul to 5 g of soil/sediment is equivalent to 50 ug/kg of each surrogate and internal standard.

6.4.3.1.3 Purge and Trap Analysis

Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the sample into the purging chamber. Heat the sample to 40°C while purging for 11 (± 0.1) minutes. Proceed with the analysis as outlined in Section 6.4.1.10.

6.4.3.1.4 For matrix spike analysis of low-level soils/sediment, add 10 ul of the matrix spike solution (Section 5.7) to the 5 ml of water (Section 6.4.3.2) equivalent to 50 ug/kg of each matrix spike standard. Analyze as in Section 6.4.3.1.

6.4.3.1.5 Immediately after weighing the sample, weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be

reported relative to the dry weight of sediment.

$$\frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100 = \% \text{ moisture}$$

6.4.3.2 Medium-Level Soil Method

If saturated peaks occurred or would occur when a 1 g sample was analyzed by the low-level method, the medium-level method must be used. The medium-level soil method is based on extracting 4 g of soil/sediment sample with 10 ml of methanol (9mL of methanol and 1 mL surrogate), and add 100 ul of the methanol extract to reagent water containing the internal standards. This is purged at ambient temperature.

6.4.3.2.1 The GC/MS system should be set up as in Sections 6.4.1.1 - 6.4.1.4. This should be done prior to the addition of the methanol extract to reagent water. Initial and continuing calibrations (Section 6.2.1.5) are performed by adding standards to reagent water and purging at ambient temperature, just as aqueous calibrations are performed.

6.4.3.2.2 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) into a tared 15 ml vial. Use a top-loading balance. Note and record the actual weight to the nearest 0.1 g. Determine the % Moisture as in Section 6.4.3.1.5.

6.4.3.2.3 Quickly add 9.0 mL of methanol to the vial. Then add 1.0 mL of the system monitoring compound spiking solution to the vial. Cap and shake for 2 minutes. NOTE: The steps in paragraphs 6.4.3.2.1 to 6.4.3.2.3 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a

laboratory free of solvent fumes.

6.4.3.2.4 Using a disposable pipette, transfer approximately 1 ml of extract into a GC vial for storage. The remainder may be disposed of. Transfer approximately 1 ml of the reagent methanol to a GC vial for use as the method blank for each case or set of 20 samples, whichever is more frequent. These extracts may be stored in the dark at 4°C (± 2°C) prior to analysis.

6.4.3.2.5 The addition of a 100 ul aliquot of the extracts into 5 ml of reagent water will give a concentration equivalent of 6,200 ug/kg of each system monitoring compound standard.

6.4.3.2.6 The following table can be used to determine the volume of methanol extract to add to the 5 ml of reagent water for analysis. From the estimated concentration, determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-level analysis to determine the appropriate volume. If the sample was submitted as a medium-level sample, start with 100 ul. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of linear range of the curve.

Estimated Concentration Range (ug/kg)	Volume Methanol Extract (ul)
500 - 10,000	100
1,000 - 20,000	50
5,000 - 100,000	10
25,000 - 500,000	100 of 1/50 dilution

6.4.3.2.7 Calculate appropriate dilution factor for concentrations exceeding the table. Dilute a aliquot of the methanol extract and take 100 ul for analysis. The volume of methanol in the 5 ml syringe must be constant. Therefore, add to the 5 ml syringe whatever volume of methanol is necessary to maintain a volume of 100 ul added to the syringe.

6.4.3.2.8 Remove the plunger from a 5 ml syringe and fill until overflowing with reagent water. Replace the plunger and adjust the volume to 4.9 ml. Pull the plunger back to 5 ml to allow volume of the addition of sample and standards. Add 10 ul of the internal standard solution. Also add the volume of methanol extract determined in Section 6.4.3.2.6, add a volume of methanol solvent to total 100 ul (excluding methanol in standards).

6.4.3.2.9 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

6.4.3.2.10 Proceed with the analysis as outlined in Sections 6.4.1.10 - 6.4.1.12. Analyze all reagent blanks on the same instrument as the samples. the standards should also contain 100 ul of methanol to simulate the sample conditions.

6.4.3.4.11 For a matrix spike in medium-level sediment/soil samples, add 8 ml of methanol, 1 ml of surrogate spike solution (Section 5.3.3.1), and 1 ml of matrix spike solution (Section 5.7.3). This results in a 6,200 ug/kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100 ul aliquot of this extract to 5 ml of water for purging (as per Section 6.4.3.2.2).

6.5 Data Interpretation

6.5.1 Qualitative Analysis

6.5.1.1 An analyte (e.g., those listed in Table 4-1) is identified by comparison of the sample mass spectrum with a standard reference spectrum obtained on the user's GC/MS. The standard reference spectra are obtained from the analysis of calibration standards. Two criteria must be satisfied to verify identification: (1) Elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component

and the standard component mass spectra.

6.5.1.2 For establishing correspondence of the GC RRT, the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles.

6.5.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

6.5.1.3.1 All ions present in the standard mass spectra at a relative intensity >10% (most abundant ion in the sample spectrum) must be present in the sample spectrum.

6.5.1.3.2 The relative intensities of ions specified in Section 6.5.1.3.1 must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30% and 70%.).

6.5.1.3.3 Ions >10% in the sample spectrum, but not present in the standard spectrum, must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra for CLP analyses. For all compounds below the method reporting limit, the actual value is followed by a "J", e.g., "3J".

6.5.1.4 If a compound cannot be verified by all of the criteria in Section 6.5.1.3 but, in the technical judgement of the Mass Spectral Interpretation Specialist the identification is correct, the analyst shall report that identification and

proceed with quantification in Section 6.5.2.

6.5.1.5 A library search is executed for non-target sample components for the purpose of tentative identification. For this purpose, the 1989 (or more recent) release of the NIST/EPA/MSDC mass spectral library, containing 50,000 spectra, is used.

6.5.1.5.1 Up to 10 non target organic compounds of greatest apparent concentration excluding the system monitoring compounds, are tentatively identified via a forward search of the NIST/EPA/MSDC Library. After visual comparison of sample spectra with the nearest library searches the mass spectral interpretation specialist assigns a tentative identification.

6.5.1.5.2 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

6.5.1.5.3 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30% and 70%.)

6.5.1.5.4 Molecular ions present in reference spectrum should be present in sample spectrum.

6.5.1.5.5 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

6.5.1.5.6 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination of coeluting compounds. Data system

library reduction programs can sometimes create these discrepancies.

6.5.1.6 If, in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e. unknown aromatic, unknown hydrocarbon, unknown acid type, or unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

6.5.2 Quantitative Analysis

6.5.2.1 Components associated with the calibration standards are quantified by the internal standard method. The internal standard used shall be that which is listed in Section 6.2.7. Use the EICP areas of the characteristic ions for analytes, listed in Table 4-3.

6.5.2.2 Internal standards responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections made as required. The EICP of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of 2 (-50% to +100%), the MS system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

6.5.2.2.1 If after reanalysis the EICP areas for all internal standards are inside the contract limits (-50% to +100%), the problem with the first analysis is considered to have been

within the control of the laboratory. The data from the reanalysis with the EICP's within the QC limits is considered the initial analysis and is reported as such on all data deliverables.

6.5.2.2.2 If the reanalysis of the sample does not solve the problem, i.e., the EICP areas are outside the QC limits for both analyses, submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. Document in the Case Narrative all inspection and corrective actions taken.

6.5.2.3 The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. Use the RRF as determined in Section 6.2.7 and the equations below.

6.5.2.3.1 Water

$$\text{Concentration (ug/l)} = \frac{(A_x)\,(I_s)\,(D_f)}{(A_{is})\,(RRF)\,(V_o)}$$

Where:

A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion for the specific internal standard from Exhibit E

I_s = amount of internal standard added in ng

V_o = volume of water purged in ml

D_f = Dilution factor.

6.5.2.3.2 Sediment/Soil (Medium-Level)

$$\text{Concentration (ug/kg)} = \frac{(A_x)\,(I_s)\,(V_t)}{(A_{is})\,(RRF)\,(V_i)\,(W_s)\,(D)}$$

6.5.2.3.3 Sediment/Soil (Low-Level)

$$\text{Concentration (ug/kg) (dry weight basis)} = \frac{(A_x)(I_s)}{(A_{is})(RRF)(W_s)(D)}$$

Where:

A_x, I_s, A_{is} = same as for water, above

V_t = volume of total extract (ul) (Use 10,000 ul or a factor of this when dilutions are made.)

V_i = volume of extract added (ul) for purging

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

W_s = weight of sample extracted (g) or purged

6.5.2.3.4 An estimated concentration is calculated for tentatively identified compounds. For quantification, the nearest internal standard free of interferences is used.

The formula for calculating concentrations is the same as in Section 6.5.2.3. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. An RRF of 1 is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively-identified compounds, as well as those identified as unknowns.

6.5.2.3.5 Xylenes (o-, m-, and p-isomers) are to be reported as xylenes (total). If o- and p-xylene overlap, the xylenes must be quantitated as m-xylene. The concentration of all xylene isomers must be added together to give the total.

6.5.2.3.6 1,2-dichloroethene (trans- and cis-

stereoisomers) are to be reported as 1,2-dichloroethene (total). The concentrations of both isomers must be added together to give the total.

6.5.2.4 Calculate system monitoring compound standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits, and report on appropriate form.

Compound	%Recovery Water	Soil
Toluene-d_8	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d_4	76-114	70-121

6.4.2.4.1 Calculation for Surrogate Recovery

$$\% \text{ Surrogate Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

Where:

Q_d = quantity determined by analysis

Q_a = quantity added to sample

6.5.2.4.2 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance.
- Reanalyze the sample if none of the above reveal a problem.

6.5.2.4.3 If the reanalysis of the sample solves the problem, the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the QC limits. This shall be considered the initial analysis and shall be reported as such on all data deliverables.

6.5.2.4.4 If the reanalysis of the sample does not solve the problem, i.e., system monitoring compound recoveries are outside the QC limits for both analyses, submit the surrogate spike recovery data and the sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables.

6.5.2.4.5 If the sample with system monitoring compound recoveries outside the limits is the sample used for the matrix spike and matrix spike duplicate and the surrogate recoveries of the matrix spike and matrix spike duplicate show the same pattern (i.e., outside the limits), the sample, matrix spike, and matrix spike duplicate do not require reanalysis. Document in the Case Narrative the similarity in surrogate recoveries.

6.5.2.5 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, for the following, whichever is most frequent:

- Each Case of field samples received, OR
- Each 20 field samples in a Case, OR
- Each group of field samples of a similar concentration level (soils only), OR
- Each 14 calendar day period (7 calendar day period for 14-day turnaround contracts) during which field samples in a Case were received (said period beginning with the receipt of the first sample in that Sample Delivery Group (SDG)).

6.5.2.5.1 Calculate the concentrations of the matrix spike compounds using the same equations as for target compounds. Calculate the recovery

of each matrix spike compound as follows:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

6.5.2.5.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{|MSR - MSDR|}{(½)\ (MSR+MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

6.5.2.5.3 The limits for matrix spike compound recovery and RPD are given below. Although these limits are only advisory, an effort is made to determine the cause or reason for out of control situations.

Compounds	%Recovery Water	RPD Water	%Recovery Soil	RPD Soil
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

7.0 QUALITY CONTROL FOR COMMERCIAL CLIENTS

7.1 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the

following operations.

7.1.1 A QC check sample concentrate is required containing each analyte at a concentration of 10 ug/ml in methanol. The QC check sample concentrate may be prepared from pure standard materials or purchased as a certified solution. If prepared by the laboratory, the QC check sample concentrate must be made using stock standards prepared independently from those used for calibration.

7.1.2 Prepare a QC check sample to contain 20 ug/l of each analyte by adding 200 ul of QC check sample concentrate to 100 ml of reagent water.

7.1.3 Four 5-ml aliquots of the well-mixed QC check sample are analyzed.

7.1.4 Calculate the average recovery (x) in ug/l and the standard deviation of the recovery (s) in ug/l for each analyte using the 4 results.

7.1.5 For each analyte, compare "s" and "x" with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 4-5. If "s" and "x" for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual "s" exceeds the precision limit or any individual "x" falls outside the range for accuracy, the system performance is unacceptable for that analyte.

7.1.6 The following procedure must be used for unacceptable system performance.

7.1.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Section 7.1.2.

7.1.6.2 Beginning with Section 7.1.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 7.1.2.

7.2 The laboratory must, on an ongoing basis, analyze a reagent blank, a matrix spike, and a matrix spike duplicate for each analytical batch (up to a maximum of 20 samples/batch).

7.3 Lab control spikes (LCS) are included in each analytical batch. The LCS is used to create control charts to monitor laboratory performance. After the analysis of 5 LCS samples, calculate the average "p" and the standard deviation of the "p" (s_p). Express the accuracy assessment as a "p" interval from $p - 2s_p$ to $p + 2s_p$. If p=90% and s_p=10%, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g. after each 5 to 10 new accuracy measurements).

7.4 To determine acceptable accuracy and precision limits for LCS, the following procedure should be performed.

7.4.1 Once a minimum of 30 LCS's have been analyzed, calculate the average "p" and the "s_p" for each of the spike compounds.

7.4.2 Calculate the upper and lower control limits for method performance for each LCS compound. This should be done as follows:

Upper Control Limit (UCL) = p + 3s

Lower Control Limit (LCL) = p - 3s

7.4.3 If recovery is not within limits, the following procedures are required.

7.4.3.1 Check to be sure there are no errors in calculation, spike solutions, and internal standards. Also, check instrument performance.

7.4.3.2 Document the out-of-control situation and report it to the Lab Supervisor immediately.

7.5 Method Reporting Limit (MRL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. These limits are determined annually by analyzing 7 consecutive standards at the EPA quantitation limit (Table 4-2) and multiplying the standard deviation by 3.143.

8.0 DATA CONTROL

8.1 Sample raw data is generated from the instruments and automatically downloaded onto the Hewlett Packard data system.

8.2 Analysts check the sample criteria to ensure that the sample analysis was good. Samples are reanalyzed when criteria are out.

8.3 Analysts check computer calculations and spectral interpretations to confirm that compounds are present in the sample.

8.4 A cover sheet (Figure 4-1) is prepared by the analyst per sample detailing the:

- EPA ID,
- Ceimic ID,
- file ID,
- blank associated with the sample,
- matrix,
- volume,
- column used in analysis,
- % moisture,
- pH,
- detection limit,
- concentration, and
- recovery.

8.5 Standards and Blanks are labelled as "V" for volatiles, "S" for semivolatiles, and "P" for pesticides followed by STD for standard, "BLK" for blank, followed by the concentration of the standard or the number of the blank.

8.6 Data is reported to the two significant figures for pesticides, and volatile and semivolatile results greater than or equal to 10, volatile and semivolatile results less than 10 are reported to one significant figure.

8.7 The data qualifiers to be used are as follows:

8.7.1 **"U"**

Indicates compound was analyzed for but not detected. The sample quantitation limit must be corrected for dilution and for percent moisture. For example, 10 U for phenol in water if the sample final volume is the protocol-specified final volume. If a 1 to 10 dilution of extract is necessary, the reported limit is 100 U. For soil samples,

the value must also be adjusted for percent moisture. For example, if the sample had 24% moisture and a 1 to 10 dilution factor, the sample quantitation limit for phenol (330 U) would be corrected to

$$\frac{(330\ U)}{D} \times df \text{ where } D = \frac{100 - \%\ moisture}{100}$$

and if df = dilution factor

For example, at 24% moisture, $D = \frac{100-24}{100} = 0.76$

$$\frac{(330\ U)}{.76} \times 10 = 4300\ U$$ rounded to the appropriate number of significant figures

For soil samples subjected to GPC clean-up procedures, the extract must be concentrated to 0.5 mL, and the sensitivity of the analysis is not compromised by the cleanup procedures. Therefore, the CRQL values in Exhibit C will apply to all samples regardless of cleanup. However, if a sample extract cannot be concentrated to the protocol-specific volume, this fact must be accounted for in reporting the sample quantitation limit.

8.7.2 **"J"**

Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit but greater than zero. For example, if the sample quantitation limit is 10 ug/L, but a concentration of 3 ug/L is calculated, report it as 3J. The sample quantitation limit must be adjusted for dilution as discussed for the "U" flag.

8.7.3 **"N"**

Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds, where the identification is based on a mass spectral library search. It is applied to all TIC

results. For generic characterization of a TIC, such as chlorinated hydrocarbon, the "N" code is not used.

8.7.4 **"P"**

This flag is used for a pesticide/Aroclor target when there is greater than 25% difference for detected concentrations between two GC columns. The lower of the two values is reported on Form I and flagged with a "P".

8.7.5 **"C"**

This flag applies to pesticide results where the identification has been confirmed by GC/MS. If GC/MS confirmation was attempted but was unsuccessful, do not apply this flag, instead use a laboratory-defined flag, discussed below.

8.7.6 **"B"**

This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank confirmation and warns the data user to take appropriate action. This flag must be used for TIC as well as for a positively identified target compound.

8.7.8 **"E"**

This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis. If one or more compounds have a response greater than full scale, except as noted in Exhibit D, the sample or extract must be diluted and re-analyzed according to the specifications in Exhibit D. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form I for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate copies of Form I. The Form I for the diluted sample shall have the "DL" suffix appended to the sample number. NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range of each peak

should be considered separately, e.g., a diluted analysis is not required for total xylenes unless the concentration of the peak representing the single isomer exceeds 200 ug/L or the peak representing the two coeluting isomers on that GC column exceeds 40 ug/L. Similarly, if the two 1,2-Dichloroethene isomers coelute, a diluted analysis is not required unless the concentration exceeds 400 ug/L.

8.7.9 **"D"**

This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is re-analyzed at a higher dilution factor, as in the "E" Flag above, the "DL" suffix is appended to the sample number on the Form I for the diluted sample, and all concentration values reported on that Form I are flagged with the "D" Flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample or extract.

8.7.10 **"A"**

This flag indicates that a TIC is suspected aldol-condensation product.

8.8 Data is reviewed by the Laboratory Manager, and any errors are corrected.

8.9 All data is then reviewed by the Mass Spectra Interpretation Specialist.

8.10 At this point, data is ready to be inputted into Formaster. Refer the Data Package Preparation SOP (SOP 013).

9.0 NOTEBOOK FORMAT

9.1 All samples run in the VOA Laboratory shall be logged into the designated Instrument Run Log on a daily basis. Sample ID #'s, date of analysis, volume of sample and instrumental conditions shall be recorded (Figure 4-3).

9.2 All maintenance, performed either by the technicians or by service people, shall be recorded into the designated maintenance logs. All problems and corrective actions taken shall be recorded as well as date and initials of the technician.

9.3 Logbooks must comply with the EPA SOW criteria.

9.3.1 Sequential numbered pages are required in all logbooks.

9.3.2 Single-line cross-our/initials/date must be used to document all corrections in logbooks.

9.3.3 All dates in logbooks must include the year.

9.3.4 All logbook entries must include the initials of the analyst.

9.3.5 Unused portions of logbook pages must be lined-out.

9.3.6 The use of correction fluid or erasing is prohibited in all logbooks.

9.3.7 Any logbook containing EPA CLP information must comply completely with the above criteria. This includes entries for commercial cases.

10.0 TRACEABILITY OF STANDARDS

10.1 Standard Receipt Log

All standards obtained from commercial vendors or government sources are stored in the VOA freezer at -10°C to -20°C and are logged into the Volatile Organic Standard Receipt Log as follows (Figure 4-4).

10.1.1 Date

10.1.2 Compound or standard name

10.1.3 Receipt ID as follows: "V" for volatiles, followed by last 2 digits of the year, the month, the day of receipt, and a letter from A to Z. The standard container is labelled with this receipt ID. (Example: V911021A is the first standard received on October 21, 1991.)

10.1.4 Vendor name

10.1.5 Lot number

10.1.6 Concentration

10.1.7 Expiration date

10.1.8 Storage location

10.1.9 Analyst's initials

10.2 Stock Standards Log

Stock standards are compounds or solutions which are diluted into working standards. These stock standards may be obtained from commercial vendors or government sources or prepared from neat standards. Stock standards are logged into the Volatile Organics Stock Standards Log (Figure 4-5) as follows:

10.2.1 Date

10.2.2 Compound or standard name

10.2.3 Receipt ID

10.2.4 Initial weight/concentration

10.2.5 Total ug/ul added

10.2.6 Volume

10.2.7 Final concentration

10.2.8 Stock Standard ID as follows: "V" for volatiles, "S" for stock, followed by the last 2 digits of the year, the month, the day, and a letter from A to Z. The date refers to the date the stock ampule is opened or the date the stock standard is prepared from other stock standards. (Example: VS911021A is the first stock standard received or prepared on October 21, 1991.)

10.2.9 Storage location

10.2.10 Stock standard label, which shows exactly how the standard vial is labelled.

10.2.11 Analyst's initials

10.3 Working Standards Log

Working standards are standards or solutions used to prepare initial calibration standards, continuing calibration standards, or matrix spike samples. Quality control mixtures containing internal standards and surrogates which are spiked into all standards, blanks, and samples are also working standards. These working standards, which may be prepared from stock solutions or obtained from commercial vendors, are

stored in the VOA freezer at -10°C to -20°C and are logged into the Volatile Organics Working Standard Log as follows:

10.3.1 Working standards are assigned an entry number as follows: month, hyphen, number. The date of preparation refers to the date the working standard is prepared from stock standards or the day an ampule obtained from a commercial vendor is cracked. (Example: 10-5, the fifth working standard prepared in October.)

10.3.2 Date

10.3.3 Compound or standard name

10.3.4 Stock ID or sample receipt ID

10.3.5 The entry number for working standards is logged into the Instrument Run Log in the "Comments" section for all initial and continuing calibrations.

10.3.6 The working surrogate and internal standard solution, QC Mix, is labelled with a "Q", followed by the working standard entry number. (Example: Q10-1 is the QC Mix working standard which was the first working standard prepared in October.)

10.3.7 The QC Mix number is logged into the MS Header for each standard, sample, and blank. (Example: VS5050 50 ppb Q10-1. This continuing calibration standard was prepared using QC Mix 10-1, the first working standard prepared in October.)

11.0 EQUIVALENCY OF VOLATILE STANDARDS

11.1 To establish the reliability of calibration standards, the following procedure is employed.

11.1.1 Each lot of standard received is analyzed at 50 ppb and compared to an independent EPA-certified check standard obtained from a commercial vendor under an EPA Cooperative Research and Development Agreement.

11.1.2 The % Difference is calculated as follows:

$$\% \text{ Difference} = \frac{\text{Conc. Ceimic Std.} - \text{Check Std. Conc.}}{\text{Check Std. Conc.}}$$

11.1.3 The standards are considered to be equivalent if the % Different is $\leq$25.

11.1.4 Results are recorded in the VOA Standard Equivalency Log as follows:

11.1.4.1 Date,

11.1.4.2 standard name,

11.1.4.3 source,

11.1.4.4 lot number,

11.1.4.5 check standard name,

11.1.4.6 source,

11.1.4.7 comments, and

11.1.4.8 analyst's initials.

11.2 Documentation to verify standard integrity is maintained in each laboratory. This documentation is reviewed by the laboratory supervisor, and includes weighing logbooks, calculations, chromotographs and mass spectra produced by the laboratory or by chemical supply houses.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument maintenance is performed only by the GC/MS Manager and can include cleaning the source, changing the trap, changing the ferrule if a leak is suspected, and changing a column. All analysts must refer to the Hewlett Packard Maintenance Manual, located in the Volatile Organics Laboratory, for proper technique. In addition, each analyst must take a Hewlett Packard seminar on cleaning the source.

TABLE 4-1
RETENTION TIME AND CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

Compound	Retention Time (min) CAP	Packed	Primary Ion	Secondary Ion(s)
Acetone	7.06	5.99	43	58
Benzene	13.18	16.31	78	52, 77
Bromochloromethane (I.S.)	11.95	8.78	128	49, 130, 51
Bromodichloromethane	15.16	13.80	83	85, 129
4-Bromofluorobenzene (surr.)	20.34	27.72	95	174, 176
Bromoform	19.83	19.18	173	171, 175, 252
Bromomethane	4.73	2.02	94	96, 79
2-Butanone	11.53	11.42	72	57, 43
Carbon disulfide	7.39	6.90	76	78
Carbon tetrachloride	12.81	13.16	117	119, 121
Chlorobenzene	18.60	23.84	112	114, 77
Chlorobenzene-d_5 (I.S.)	18.56	23.70	117	82, 199
Dibromochloromethane	17.66	16.54	129	208, 206
Chloroethane	4.96	3.44	64	66, 49
Chloroform	12.14	10.88	83	85, 47
Chloromethane	3.89	1.20	50	52, 49
1,1-Dichloroethane	10.15	9.46	63	65, 83
1,2-Dichloroethane	13.20	11.56	62	64, 98
1,2-Dichloroethane-d_4 (surr.)	13.06	11.57	65	102
1,1-Dichloroethene	6.84	8.23	96	61, 98
cis-dichloroethane	11.46	10.24		
Trans-1,2-dichloroethene	9.07	10.24	96	61, 98
1,2-Dichloropropane	14.69	15.03	75	77, 39
Cis-1,3-dichloropropene	15.90	15.30	75	77,39
Trans-1,3-dichloropropene	16.76	16.58	75	77, 39
1,4-Difluorobenzene (I.S.)	13.88	23.70	114	63, 88
Ethylbenzene	18.76	25.62	106	91
2-Hexanone	17.43	21.10	43	58, 57, 100
Methylene chloride	8.29	5.49	84	49, 51, 86
4-Methyl-2-pentanone	16.13	19.55	43	58, 100
Styrene	19.56	28.81	104	78, 103
1,1,2,2-Tetrachloroethane	20.52	21.47	83	85, 131, 133
Tetrachloroethene	17.29	21.47	164	129, 131, 166
Toluene	16.43	22.65	92	91, 65
Toluene-d_8 (surr.)	16.33	22.47	98	70, 100
1,1,1-Trichloroethane	12.47	12.75	97	99, 117
1,1,2-Trichloroethane	17.03	16.63	97	83, 85, 99
Trichloroethene	14.33	15.85	130	95, 97, 132
Vinyl chloride	4.10	2.52	62	64, 61
O-Xylene	19.61	30.54	106	91
M-Xylene	19.00	29.68		
P-Xylene	19.00	30.54		

TABLE 4-2
TARGET COMPOUND LIST (TCL) AND QUANTITATION LIMITS FOR EPA CONTRACT WORK
(Refer to Section 7.5 for Commercial Limits)

Volatiles	CAS Number	Quantitation Limits* Water (ug/l)	Soil Low (ug/kg)	Soil Med
1. Chloromethane	74-87-3	10	10	1200
2. Bromomethane	74-83-9	10	10	1200
3. Vinyl chloride	75-01-4	10	10	1200
4. Chloroethane	75-00-3	10	10	1200
5. Methylene chloride	75-09-2	10	10	1200
6. Acetone	67-64-1	10	10	1200
7. Carbon disulfide	75-15-0	10	10	1200
8. 1,1-Dichloroethene	75-35-4	10	10	1200
9. 1,1-Dichloroethane	75-34-3	10	10	1200
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200
11. Chloroform	67-66-3	10	10	1200
12. 1,2-Dichloroethane	107-06-2	10	10	1200
13. 2-Butanone	78-93-3	10	10	1200
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200
15. Carbon tetrachloride	56-23-5	10	10	1200
16. Bromodichloromethane	75-27-4	10	10	1200
17. 1,2-Dichloropropane	78-87-5	10	10	1200
18. Cis-1,3-dichloropropene	10061-01-5	10	10	1200
19. Trichloroethene	79-01-6	10	10	1200
20. Dibromochloromethane	124-48-1	10	10	1200
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200
22. Benzene	71-43-2	10	10	1200
23. Trans-1,3-dichloropropene	10061-02-6	10	10	1200
24. Bromoform	75-25-2	10	10	1200
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200
26. 2-Hexanone	591-78-6	10	10	1200
27. Tetrachloroethene	127-18-4	10	10	1200
28. Toluene	108-88-3	10	10	1200
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200
30. Chlorobenzene	108-90-7	10	10	1200
31. Ethyl benzene	100-41-4	10	10	1200
32. Styrene	100-42-5	10	10	1200
33. Xylenes (total)	1330-20-7	10	10	1200

* wet weight basis

TABLE 4-3
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS *

Parameter	Primary Ion**	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	45, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43***	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
Trans-1,3-dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	--
Cis-1,3-dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total xylenes	106	91

* See Table 4-1 for internal standard and system monitoring compounds.

** The primary ion should be used unless interferences are present, in which case a secondary ion may be used.

*** m/z 72 must be present for positive identification.

TABLE 4-4
CALIBRATION AND QC ACCEPTANCE CRITERIA*

Parameter	Range for Q (ug/l)	Limit for s (ug/l)	Range for x (ug/l)	Range p, p_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
Chloroethane	7.3-32.4	11.4	8.4-40.4	14-230
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
Trans-1,2-dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
Cis-1,3-dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
Trans-1,3-dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/l
s = Standard deviation of 4 recovery measurements, in ug/l
x = Average recovery for 4 recovery measurements, in ug/l
p, p_s = % Recovery measured
D = Detected; result must be >0

* Criteria from 40 CFR Part 136 for Method 624 were calculated assuming a QC check sample concentration of 20 ug/l.

TABLE 4-5
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Parameter	Accuracy, as Recovery, x' (ug/l)	Single Analyst Precision, s_r' (ug/l)	Overall Precision, S' (ug/l)
Benzene	0.93C+2.00	0.26x-1.74	0.25x-1.33
Bromodichloromethane	1.03C-1.58	0.15x+0.59	0.20x+1.13
Bromoform	1.18C-2.35	0.12x+0.34	0.17x+1.38
Bromomethane	1.00C	0.43x	0.58x
Carbon tetrachloride	1.10C-1.68	0.12x+0.25	0.11x+0.37
Chlorobenzene	0.98C+2.28	0.16x-0.09	0.26x-1.92
Chloroethane	1.18C+0.81	0.14x+2.78	0.29x+1.75
2-Chloroethylvinyl ether	1.00C	0.62x	0.84x
Chloroform	0.93C+0.33	0.16x+0.22	0.18x+0.16
Chloromethane	1.03C-1.81	0.37x+2.14	0.58x+0.43
Dibromochloromethane	1.01C-0.03	0.17x-0.18	0.17x+0.49
1,2-Dichlorobenzene[b]	0.94C+4.47	0.22x-1.45	0.30x-1.20
1,3-Dichlorobenzene	1.06C+1.68	0.14x-0.48	0.18x-0.82
1,4-Dichlorobenzene[b]	0.94C+4.47	0.22x-1.45	0.30x-1.20
1,1-Dichloroethane	1.05C+0.36	0.13x-0.05	0.16x+0.47
1,2-Dichloroethane	1.02C+0.45	0.17x-0.32	0.21x-0.38
1,1-Dichloroethene	1.12C+0.61	0.17x+1.06	0.43x-0.22
Trans-1,2-dichloroethene	1.05C+0.03	0.14x+0.09	0.19x+0.17
1,2-Dichloropropane[a]	1.00C	0.33x	0.45x
Cis-1,3-dichloropropene[a]	1.00C	0.38x	0.52x
Trans-1,3-dichloropropene[a]	1.00C	0.25x	0.34x
Ethyl benzene	0.98C+2.48	0.14x+1.00	0.26x-1.72
Methylene chloride	0.87C+1.88	0.15x+1.07	0.32x+4.00
1,1,2,2-Tetrachloroethane	0.93C+1.76	0.16x+0.69	0.20x+0.41
Tetrachloroethene	1.06C+0.60	0.13x-0.18	0.16x-0.45
Toluene	0.98C+2.03	0.15x-0.71	0.22x-1.71
1,1,1-Trichloroethane	1.06C+0.73	0.12x-0.15	0.21x-0.39
1,1,2-Trichloroethane	0.95C+1.71	0.14x+0.02	0.18x+0.00
Trichloroethene	1.04C+2.27	0.13x+0.36	0.12x+0.59
Trichlorofluoromethane	0.99C+0.39	0.33x-1.48	0.34x-0.39
Vinyl chloride	1.00C	0.48x	0.65x

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/l
s_r' = Expected single analyst standard deviation of measurements at an average concentration of x, in ug/l
X' = Expected inter-laboratory standard deviation of measurements at an average concentration found in x, in ug/l
C = True value for the concentration, in ug/l
x = Average recovery found for measurements of samples containing a concentration of C, in ug/l
[a] - Estimates based upon the performance in a single laboratory
[b] - Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.

TABLE 4-6

SYSTEM MONITORING COMPOUND SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Low/Medium Water	Low/Medium Soil/Sediment
4-Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d_4	76-114	70-121
Toluene-d_8	88-110	84-138

FIGURE 4-1
VOA

EPA ID_____
CEIMIC ID_____
FILE_____

74-87-3-----Chloromethane_____
74-83-9-----Bromomethane_____
75-01-4-----Vinyl chloride_____
75-00-3-----Chloroethane_____
75-09-2-----Methylene chloride_____
67-64-1-----Acetone_____
75-15-0-----Carbon disulfide_____
75-35-4-----1,1-Dichloroethene_____
75-34-3-----1,1-Dichloroethane_____
540-59-0----1,2-Dichloroethene(total)_____
67-66-3-----Chloroform_____
107-06-2----1,2-Dichloroethane_____
78-93-3-----Butanone_____
71-55-6-----1,1,1-Trichloroethane_____
56-23-5-----Carbon tetrachloride_____
75-27-4-----Bromodichloromethane_____
78-87-5-----1,2-Dichloropropane_____
10061-01-5--Cis-1,3-dichloropropene_____
79-01-6-----Trichloroethene_____
124-48-1----Dibromochloromethane_____
79-00-5-----1,1,2-Trichloroethane_____
124-43-1----Dibromochloromethane_____
79-00-5-----1,1,2-Trichloroethane_____
71-43-2-----Benzene_____
10061-02-6--Trans-1,3-dichloropropene_____
75-25-2-----Bromoform_____
108-10-1----4-Methyl-2-pentanone_____
591-78-6----2-Hexanone_____
127-18-4----Tetrachloroethene_____
79-34-5-----1,1,1,2-Tetrachloroethane_____
108-88-3----Toluene_____
108-90-7----Chlorobenzene_____
100-41-4----Ethylbenzene_____
100-42-5----Styrene_____
1330-20-7---Xylene_____

ASSOCIATED BLANK_____ % MOISTURE_____
DATE RECEIVED_____ pH_____
DATE EXTRACTED_____ DETECTION LIMIT_____
LEVEL_____ CONC._____
MATRIX_____ RECOVERY_____
VOLUME_____ DF_____
COLUMN_____ DRY FACTOR_____

CEMIC CORPORATION – VOLATILE ORGANICS LABORATORY

pH ANALYSIS LOGBOOK

DATE	TIME	CEIMIC ID#	EPA ID#	pH (Paper)	INITIALS

Figure 4–2

CEIMIC CORPORATION
VOLATILE ORGANICS LABORATORY

MS#: _____

INSTRUMENT RUN LOG/INSTRUMENT TRACKING LOG

Column: _____

FRN#	ARCH TAPE	CH #	SAMPLE ID	CONC /VOL	E.M. VOLT	INIT	DATE		COMMENTS
>									
>									
>									
>									
>									
>									
>									
>									
>									
>									
>									
>									
>									

Figure 4–3

CEIMIC CORPORATION
VOLATILE ORGANICS LABORATORY

Standard Receipt Log

Receipt Date	Compound Name	Receipt ID*	Received From	Lot Number	Conc. or Purity	Expiration Date	Storage Location	Initial

* Receipt ID Example: V–890104A
V = Volatiles
A = 1st bottle received on this day

** Label bottle with Receipt ID

Figure 4–4

CEIMIC CORPORATION
VOLATILE ORGANICS LABORATORY

Stock Standards Log

Date	Compound Name	Receipt ID*	Initial Wt.	Final Wt.	Total ug Added	Volume (ml)	Final Conc (ug/ul)	Stock ID*	Storage Location	Labeled	Initial

* Stock ID Example: V–S890104A
V = Volatiles
S = Stock Standard
A = 1st bottle received on this day

Figure 4–5

APPENDIX C

PERTINENT SECTIONS OF CEIMIC CORPORATION'S LABORATORY QUALITY ASSURANCE PLAN

4.0 Quality Assurance Objectives for Measurement of Data in Terms of Precision, Accuracy, Representativeness, Completeness, and Comparability

4.1 Quality Assurance Objectives - Definitions

As part of the evaluation component of the QA Program, laboratory results are compared with certain data quality objectives. These objectives, in terms of precision, accuracy, representativeness, completeness and comparability, are defined as follows:

Precision - The agreement or reproducibility among individual measurements of the same property, usually made under the same conditions.

Accuracy - The degree of agreement of a measurement with the true or accepted value.

Representativeness - The degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition.

Completeness - A measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct normal conditions.

Comparability - An expression of the confidence with which one data set can be compared with another data set in regard to the same property.

Quality assurance objectives vary according to the specific project and the parameters requested. The accuracy, precision and representativeness of data are functions of the origins of the samples, the procedures used to analyze samples and generate data, and the specific sample matrices. Quality control practices used in the evaluation of these data quality objectives may include blanks, replicates, spikes, lab control standards, check samples, internal standard and surrogate recoveries.

4.2 Precision and Accuracy

For each parameter analyzed, the QA objectives for precision and accuracy are determined from: 1) Published historical data; 2) method validation studies; 3) Ceimic experience with similar samples; and/or 4) project-specific requirements.

4.3 Representativeness

The representativeness of the data depends largely on the sampling procedures, but also depends on the procedures used in processing the samples. The objective for representativeness is to provide data of the same high quality as other analyses of similar samples using the same methods during the same time period within the laboratory. Representativeness can be determined for this objective by a comparison of the quality control data for these samples against other data for similar samples analyzed at the same time. Differences within 20% are acceptable.

4.4 Completeness

Completeness of an analysis is documented by including in the report sufficient information to allow the data user to assess

the quality of the results. The objective for completeness is 100% in most cases and includes: Analysis of all samples; generation and analysis of all required QC samples; sufficient documentation of associated calibration, tuning and standardization to meet the data quality objectives of the project, and records of data reduction processes. Completeness is ensured by assigning a staff member to provide a final review of the data package.

4.5 Comparability

The results of analyses can be compared with the same analyses conducted by other laboratories. The objectives for comparability are: to demonstrate traceability of standards to the National Institute of Standards and Technology (NIST) or EPA sources, to use standard methodology, to apply appropriate levels of quality control within the context of the Laboratory Quality Assurance Program, and to participate in inter-laboratory studies to document laboratory performance.

By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The QA Program documents internal performance, and the inter-laboratory studies document performance compared to other analysts at other locations.

6.0 Sample Custody

6.1 Chain-of-Custody

Samples are physical evidence collected from a facility or the environment. In hazardous waste investigations, sample data may be used as evidence in EPA enforcement proceedings. In support of potential litigation, laboratory chain-of-custody procedures have been established to ensure sample traceability from the time of receipt through completion of analysis.

The National Enforcement Investigations Center (NEIC) of the EPA considers a sample in custody under the following conditions:

- It is in your actual possession;
- it is in your view, after being in your physical possesion.
- it was in your possession and then you locked or sealed it to prevent tampering; or
- it is in a secure area.

Chain-of-custody documentation accompanies the samples as they are moved from the field to the laboratory, with shipping information and appropriate signatures indicating custody changes along the way. A Chain-of-Custody Record is included as Figure 6-1.

Laboratory chain-of-custody is initiated as samples are received and signed for by the Sample Custodian. Documentation of sample location continues as samples are signed in and out of the central storage facility for analysis using the Sample

Control Record (Figure 6-2). After analysis, extracts and any remaining samples are held in the central storage area under custody until disposal. Prior to disposal of the samples, tags and other identification are removed from the containers and placed in the project file.

6.2 Laboratory Security

The laboratory area is designated as a secure area and the doors to this area are kept locked at all times and may be accessed only by key. Authorized personnel only are allowed to enter the secure area. Visitors to the laboratories must be accompanied by Ceimic staff members.

Samples are kept within the secure laboratory area during all stages of tenure, including preparation, analysis, and storage.

6.3 Duties and Responsibilities of the Sample Custodian

Duties and responsibilities of the Sample Custodian shall include, but not be limited to, the following:

- Receiving samples
- Inspecting sample shipping containers for presence/absence and condition of:
  - o custody seals, locks, "evidence tape," etc.
  - o container breakage and/or container integrity.
- Recording condition of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks. This also includes recording whether the samples were cool upon arrival if required.

- Signing appropriate documents shipped with samples (i.e., airbills, chain-of-custody forms, traffic reports, etc.).
- Verifying and recording agreement or non-agreement of information on sample documents (i.e. sample tags, chain-of-custody records, traffic reports, airbills, etc.) in appropriate logbooks or on appropriate forms. If there is non-agreement, recording the problems, contacting the client for direction, and notifying appropriate laboratory personnel.
- Initiating the paperwork for sample analyses on appropriate laboratory documents and establishing project files according to laboratory SOP's.
- Marking or labelling samples with laboratory sample numbers, and cross-referencing laboratory numbers to client IDs and sample tags, as appropriate.
- Placing samples, sample extracts, and spent samples into appropriate storage and/or secure areas.
- Controlling access to samples in storage and assuring that laboratory Standard Operating Procedures are followed when samples are removed from and returned to storage.
- Assuring that sample tags are removed from the sample containers and included in the appropriate file if applicable.
- Monitoring storage conditions for proper

refrigeration temperature and prevention of cross-contamination.

- Returning shipping containers to the proper sampling teams.
- Preparing and shipping sampling kits including shipping containers, sample bottles, preservatives, labels, chain-of-custody forms and sampling instructions to clients who request them.

6.4 Sample Receipt

Sample shipments are received at Ceimic by the designated Sample Custodian. Shipping information is recorded in the Incoming Logbook and the paperwork filed chronologically. The shipping containers are then inspected and opened by the Sample Custodian, who records the following information on the Sample Log-in Sheet (Figure 6-3) as he unpacks the coolers:

- Condition of custody seal (intact, broken, absent) and shipping container
- presence of chain-of-custody records
- traffic reports
- airbills or bills of lading documenting shipment of samples
- sample tags if applicable
- Condition of sample bottles
- Sample tag ID numbers <u>not</u> recorded on chain-of-custody records or packing list
- Verification of agreement or non-agreement of information on receiving documents

- Resolution of problems or discrepancies with the client

Following resolution of any problems or discrepancies, the Sample Custodian signs the Sample Receipt Form and originates a project file for the set of samples, including in it the Sample Receipt Form.

When the Sample Custodian is not available off-hours to receive samples, the sample container is signed for by a Ceimic staff member and the time, date, and name of the person receiving the container are recorded in the Incoming Log, along with the appropriate shipping information. The samples are then stored under refrigeration in the sample receipt area, which is located within the secured area. The samples are officially received and documented by the Sample Custodian or designee on the next business day.

6.5 Sample Log-in and Identification

6.5.1 Sample Log-In

The sample log-in system consists of computerized entry into the Sample Receipt Log. The information recorded includes:

- Ceimic sample identification number,
- date of receipt,
- client name,
- client sample identification,
- sample matrix,
- analyses and methods required,
- project number,

- due date and comments, and
- initials of Sample Custodian or designee.

6.5.2 Sample Identification

In order to maintain sample identity, each sample received at Ceimic is assigned a unique chronological sample identification. Ceimic Sample ID Numbers appear in the following format:

ww - xxxx - yy - zzz

where:

"ww" - the last two digits of the current year;

"xxxx" -a four-digit project number which is assigned sequentially when a sample group is received at Ceimic;

"yy" -the sample number within the group and

"zzz" -an individual laboratory code e.g. - 93 - 0014 - 06 - ABC

The Sample Custodian assigns each sample a "ww-xxxx-yy" identification number. The "zzz" suffixes are assigned within the individual laboratories and vary from one laboratory to another.

The Ceimic Sample ID Numbers are recorded on the Sample Log-in Sheet, in the Sample Receipt Log, and on the Log-In Information Form (Figure 6-4), where they are cross-referenced with other

client identifiers.

The sample custodian clearly labels each container with its Sample ID Number. The same ID Number is placed on each sample preparation container and extract vial associated with the sample.

6.5.3 Sample Information

After completing the Sample Log-In Sheet and making entries in the Sample Receipt Log, the Sample Custodian prints out sample data on the Log-In Information Form, including:

- Client name
- Ceimic project number
- sample matrices
- project due date
- Ceimic sample ID numbers
- client sample Identification
- analyses and methods to be performed
- provision for signature of Sample Custodian (or designee)
- date of sample log-in
- provision for signature of Project Manager

After signing and dating the Log-In Information Form, the Sample Custodian notifies the Project Manager of the arrival of the samples. The Project Manager verifies that the information on

the Sample Log-In Information Form is correct by countersigning and dating the form. The original Log-In Information Form is placed in the project file.

Copies of the Log-In Information Form are distributed to the Ceimic Project Manager, the Accounting group, and the appropriate laboratory managers. Another copy of the form may be sent to the client as confirmation of sample receipt and specified analyses.

6.6 Sample Storage

Samples at Ceimic are stored in a central storage facility within the secured area. After sample receipt and log-in procedures are completed, the Sample Custodian places the samples in their original containers under refrigeration in the sample storage area. Samples for volatile organics analysis (VOA) are stored in a segregated area. The sample storage area is for samples only; no standards or reagents are present.

Refrigerators are maintained at 4°C (+/-2°C). A daily Temperature Log (Figure 6-5) is kept for each refrigerator by the Sample Custodian.

Access to the sample storage area is controlled by the Sample Custodian, who monitors sample custody. All transfers of samples into and out of storage are documented on a laboratory chain-of-custody form, the Sample Control Record. When an analyst removes a sample for preparation and/or

analysis, he signs it out on the Sample Control Record. Similarly, he signs the sample back in upon completion or at the end of the working day.

When analysis is complete, extracts and any remaining samples are retained in the central storage area until disposal. Broken or damaged samples are promptly disposed of in a safe manner and the disposal documented.

All custody documentation is kept in custody files originated by the Sample Custodian until samples are removed from storage for disposal. At that time, the custody files are entered into the central files and the disposal of samples and extracts documented.

Chain-of-custody of a sample ensures that the sample is traceable from field collection through laboratory receipt, preparation, analysis and, finally, disposal. The primary chain-of-custody documents which may be used to locate a sample at any point in time are:

- the Chain-of-Custody Form from the field describing the origin and transportation of a sample
- the laboratory Sample Receipt Log and supporting log-in records, documenting acceptance of a sample by Ceimic
- the Ceimic sample control forms, documenting the analyst who has custody and the reason for removal of a sample from storage.

Figure 6-1
CHAIN-OF-CUSTODY RECORD

Figure 6-1
CHAIN-OF-CUSTODY FORM

C H A I N - O F - C U S T O D Y
Original Chain-of-Custody Goes to Laboratory

Proj. # Project Name

Samplers (Please Print)

Date	Time	Comp.	Grab	Sample Identification	Type of Container	# of Containers	Remarks

Relinquished By (Signature)	Date/Time	Received By (Signature)	Date/Time	Remarks:
Relinquished By (Signature)	Date/Time	Received By (Signature)	Date/Time	
Relinquished By (Signature)	Date/Time	Received By (Signature)	Date/Time	

CEIMIC CORPORATION, 10 Dean Knauss Drive, Narragansett, RI 02882 (401) 782-8900 FAX (401) 782-8905

7.0 Calibration Procedures and Frequency

7.1 Instrument Calibration

Instrument calibration establishes that the analytical system is functioning correctly and at a level of sensitivity sufficient to meet required detection limits. Routine calibration provides a means of rapid detection of instrument variance and possible malfunction, ensuring that data quality is maintained. Specific calibration and check procedures are given in the analytical methods referenced in Section 8.0 of this Quality Assurance Plan. Frequency of calibration and concentration of standards are determined by the cited methods and special project requirements, as well as manufacturer recommendations.

Standard calibration curves of signal response versus concentration are generated on each analytical instrument used for a project prior to analysis of samples. A calibration curve of the appropriate linear range is established for each parameter analyzed and is verified on a regular basis with check standards. In general, Ceimic adheres to the calibration criteria specified by the analytical protocols required for the project.

7.2 Calibration Frequency

Specific calibration frequencies and procedures for the major instrumentation systems are presented below.

Gas Chromatography

An initial calibration is performed using a minimum of three concentration levels for each target compound. The initial calibration is done on each quantitation column and each instrument and is repeated each time a new column is installed or other major

change in the chromatographic system takes place.

For CLP analyses, continuing calibration takes place at the beginning and once every twelve hours. The relative percent difference (% RPD) in calibration factors for each standard must not exceed 25%.

Gas Chromatography/Mass Spectrometry

Initial calibration at five different concentration levels for each analyte is carried out for each system. Recalibration takes place whenever a major change occurs in the system, such as a column change in the GC or a source cleaning of the mass spectrometer. Continuing calibrations take place every twelve hours of instrument analysis time and, for level CLP analysis, must have a % Difference of 25% or less in response factors for calibration check compounds. Prior to analysis of any samples, GC/MS systems are tuned to method specifications for BFB and DFTPP for volatile and semi-volatile analyses, respectively. Verification of tuning criteria occurs every twelve hours of instrument run time, or as specified by the proper analytical protocol.

Inductively Coupled Plasma

Mixed standards are used to perform the initial multi-level calibration. Calibration check standards are analyzed every ten samples to verify instrument calibration. If the signal response of the check standard deviates by more than 10% from the initial calibration, the instrument is recalibrated. An interference control standard is run at the beginning and end of every analytical run.

Graphite Furnace Atomic Absorption

Several concentrations of individual standards are analyzed to establish the initial calibration curve for each metal. A calibration check standard is analyzed at the beginning and end of every analytical run and after every ten samples to verify the initial calibration of the instrument. If a check standard falls outside the control limit of +/- 10% from the initial calibration, the instrument is recalibrated. Calibration blanks are analyzed at the beginning and end of every analytical run and after every calibration check standard during the analysis.

7.3 Source and Preparation of Standards and Reagents

Primary sources of standard reference materials used for calibration, calibration checks and accuracy control are the EPA and National Institute of Standards and Technology (NIST) repositories. Reliable commercial manufacturers with CRADA and/or A2LA registration provide a secondary source of reference materials. Certain projects, especially those involving pesticide registration, may necessitate the use of reference materials supplied by the client. New standards are routinely validated against known standards that are traceable to EPA or NIST reference materials, if possible.

Reagents used in the preparation of matrix spike, surrogate standard, and internal standard spiking solutions for DQO level 4 analyses are validated using standards obtained directly from the EPA or repository traceable to it.

Reagents that are produced and used in large quantities, such as solvents, are examined for purity by subjecting an aliquot to

analysis prior to purchase of an entire lot or shipment. If the material appears satisfactory, the manufacturer or supplier is requested to set aside a sizeable portion of the same lot for Ceimic to be shipped upon request.

Quality Control Check Samples from the EPA-EMSL Quality Assurance Branch in Cincinnati, from CRADA and A2LA vendors, and from NIST are analyzed by the Ceimic laboratories.

Standards are periodically analyzed for concentration changes and visually inspected for signs of deterioration, such as color change and precipitate formation. A Standard Preparation Logbook, which contains all pertinent information regarding the source and preparation of each analytical standard is maintained in each of the Ceimic laboratories.

8.0 Analytical Procedures

The analytical methods used by Ceimic are contained in the following references:

- U.S. EPA Office of Solid Waste SW-846 "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods."
- EPA EMSL-CI 600/4-79-020 "Methods for Chemical Analysis of Water & Wastes."
- EPA Contract Lab Program "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," OLM 01.9
- EPA Contract Lab Program "Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration," ILM 03.0
- U.S. EPA Office of Drinking Water, "Methods for the Determination of Organic Compounds in Drinking Water."
- American Society for Testing and Materials "Annual Book of ASTM Standards."
- American Public Health Association, "Standard Methods for the Examination of Water and Wastewater."

The specific method numbers are presented in table 8-1.

Table 8-1

ANALYTICAL METHODS

ANALYTICAL METHODS

Parameter	Method				
INORGANICS					
	EPA	Drinking Water	SW846	ASTM Method	Standard Methods
Acidity	305.1				
Alkalinity	310.1				
Ammonia (as N)	350.2				
Biochemical Oxygen Demand (BOD)	405.1				5210 B
Bromide					4500–Br^- B
Chemical Oxygen Demand (COD)	410.4				5220 D
Chloride	325.3		9252		4500–Cl^- B
Chlorine (Total Residual)	330.5				4500–Cl G
Color	110.2				2120 B
Cyanide (Total & Amenable)	335.2		9010		4500–CN^- C
Fluoride	340.2				4500–F^- C
Hardness					2340 B
Kjeldahl and Organic Nitrogen	351.3				4500–N_{org} C
Chromium, Hexavalent			7196		3500–Cr D
Mercury	245.2				
Metals (Except Chromium VI & Hg)	200 Series 200.7		7000 Series 6010		
Nitrate–Nitrite (NO_3 – NO_2)	353.3				4500–NO_3^- E
Nitrite (NO_2–N)	353.3				
Oil & Grease	413.1		9070		
Orthophosphate	365.2				4500–P E
Oxygen, Dissolved	360.1,360.2				
pH	150.1		9040/9045		

ANALYTICAL METHODS

Parameter	Method

INORGANICS

	EPA	Drinking Water	SW846	ASTM Method	Standard Methods
Phosphate (Total)	365.2				4500–P E
Phenols (Total)	420.1		9065		
Solids (Total) TS	160.3				2540 B
Solids (Filterable) – TDS	160.1				2540 C
Solids (Non–Filterable) TSS	160.2				2540 D
Solids (Settleable)	160.5				2540 F
Solids (Volatile) – TVS	160.4				2540 E
Specific Conductance	120.1		9050		2510 B
Sulfate	375.4		9038		4500–SO_4^{2-} E
Sulfide	376.2		9030		4500–S^{2-} D
Sulfite	377.1				4500–SO_3^{2-} B
Surfactants (MBAS)	425.1				5540 C
Turbidity	180.1				2130 B

ORGANICS

	EPA	Drinking Water	SW846	ASTM Method	Standard Methods
Purgeable Halocarbons	601,624		8010,8240		
Purgeable Aromtic Hydrocarbons	602,624		8020,8240		
Acrolein & Acrylonitrile	603,624		8030,8240		
Volatile Organics	624	524.2	8240,8260		
Nonhalogenated Volatile Organics			8015,8240		
Phenol	604,625		8040		
EDB & DBCP		504			

ANALYTICAL METHODS

Parameter	Method

ORGANICS

	EPA	Drinking Water	SW846	ASTM Method	Standard Methods
Polynuclear Aromatic Hydrocarbons (PAH)	610		8100,8270		
Pesticides, PCBs	608	508	8080		
Herbicides	615	515.1	8150		
Semivolatile Organics	625	525.1	8270		
Total Organic Carbon (TOC)	415.1		9060		
Total Organic Halogens (TOX) or (EOX in soil)			9020		
Total Petroleum Hydrocarbons (TPH)	418.1		8015B	3328	

HAZARDOUS WASTE

	EPA	Drinking Water	SW846	ASTM Method	Standard Methods
TCLP Extraction			1311		
TCLP Volatiles			8240		
TCLP Semivolatiles			8270		
TCLP Pesticides			8080		
TCLP Herbicides			8150		
TCLP Metals			6010		
Ignitability			1010		
Corrosivity (pH)			9040/9045		
Reactivity			S 7.3.3.2, 7.3.4.1		

10.0 Laboratory Quality Control

10.1 Laboratory Quality Control Checks

Ceimic quality control procedures are determined by the data quality objectives (DQO) for the project. Five general levels of DQO options, Levels 1 through 5, are described in the EPA "Data Quality Objectives for Remedial Response Activities Development Process." It is the client's responsibility to specify the data quality objectives or level of quality control required to support the decision-making for his or her projects. Ceimic routinely performs analyses using quality control protocols equivalent to Level 3 (and Level 2 deliverables). As an option, Ceimic provides multiple levels of QC, based on the type of site being investigated, the level of accuracy and precision required, and the intended use of the data. The quality control procedures are derived from four primary sources:

1. Analytical methods, listed in table 8-1 of this QAP,
2. Project-specific Requirements,
3. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019).
4. Standards for Good Laboratory Practice.

For those projects requiring innovative techniques, Ceimic will recommend and implement, subject to client approval if required, the QC measures necessary to produce data of known quality.

The general QC Protocols for each laboratory, based on the levels of data quality objectives, are presented in Table 10-1.

Table 10-1

General QC Protocols

QUALITY CONTROL DATA QUALITY OBJECTIVES GC/MS	DQO Level I	DQO Level II	DQO Level III	DQO Level IV	DQO Level V
Surrogate Recoveries		X	X	X	X
MS, 1/20 Matrix & Project – specific *			X	X	
MSD, 1/20 Matrix & Project – specific *			X	X	
Method Blanks	X	X	X	X	X
Instrument Performance Check			X	X	
Initial Calibration			X	X	
Continuing Calibration			X	X	
Internal Standards			X	X	
Holding Blanks			X	X	
Lab Control Samples		X	X	X	X
Check Samples				X	
BFB and DFTPP Tuning, every 12 hours				X	

* if requested

ROBERT F. CAMARA
CHIEF FINANCIAL OFFICER

Mr. Camara is Ceimic's Chief Financial Officer and has twenty years of financial management experience.

EDUCATION:

PROVIDENCE COLLEGE
M.B.A.

PROVIDENCE COLLEGE
B.S. Business Administration

EXPERIENCE:

1993 - Present	CEIMIC CORPORATION Narragansett, Rhode Island Chief Financial Officer
1983 - 1993	RHODE ISLAND HOSPITAL TRUST NATIONAL BANK Providence, Rhode Island Vice President Corporate Account Manager
1981 - 1982	RHODE ISLAND HOSPITAL TRUST NATIONAL BANK Providence, Rhode Island Credit Analyst - Loan Administration, Credit Department
1977 - 1981	RHODE ISLAND HOSPITAL TRUST NATIONAL BANK Providence, Rhode Island Corporate Service Officer, Cash Management
1973 - 1977	RHODE ISLAND HOSPITAL TRUST NATIONAL BANK Providence, Rhode Island Teller & Head Teller, Community Banking Division

QUALITY CONTROL DATA QUALITY OBJECTIVES METALS	DQO Level I	DQO Level II	DQO Level III	DQO Level IV	DQO Level V
Method Blanks	X	X	X	X	X
Lab Control Sample, 1/20, Batch–specific		X	X	X	X
Initial & Continuing Calibration		X	X	X	
Postdigest Spike for ICP			X	X	
Postdigest Spike for GFAA		X	X	X	
Sample Spike, 1/20 matrix & Project – specific *			X	X	
Sample Duplicate, 1/20 matrix & Project – specific *			X	X	
Standard Addition			X	X	
ICP Interference Check Sample		X	X	X	
ICP Serial Dilutions				X	
Quarterly IDL		X	X	X	
Annual ICP Interelement Correction Factors				X	
Quarterly ICP Linear Range				X	

* if requested

QUALITY CONTROL DATA QUALITY OBJECTIVES GC					
	DQO Level I	DQO Level II	DQO Level III	DQO Level IV	DQO Level V
Surrogate Recoveries		X	X	X	X
MS, 1/20, Matrix and Project – specific *			X	X	
MSD, 1/20, Matrix and Project – specific *			X	X	
Method Blanks	X	X	X	X	X
Initial Calibration			X	X	
Continuing Verification			X	X	
Pesticide Cleanup Procedures			X	X	
Lab Control Samples		X	X	X	X

* if requested

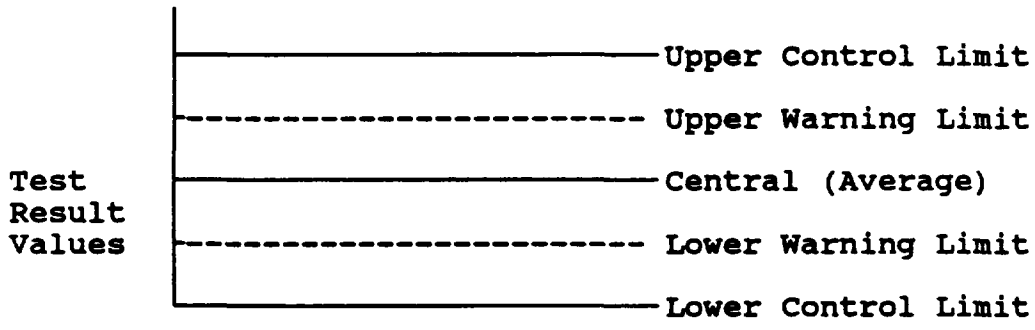
QUALITY CONTROL DATA QUALITY OBJECTIVES WET CHEMISTRY	DQO Level I	DQO Level II	DQO Level III	DQO Level IV	DQO Level V
Lab Control Sample, 1/20, Batch Specific		X	X	X	X
Matrix Spike, 1/20 Matrix & Project – specific*			X	X	
Matrix Duplicate, 1/20 Matrix & Project – specific*			X	X	
Method Blanks	X	X	X	X	X
Initial & Check Calibration		X	X	X	
Calibration Verification			X	X	
Standard Calibration Correlation			X	X	

* if requested

10.2 Control Charts

Control charts are used to monitor the variations in the analyses routinely performed by the laboratory and are intended to detect trends in these variations. Construction of a control chart requires an initial database of 20 points. As shown in Figure 10-1, the control chart limits and order of results are plotted on the horizontal scale against a vertical scale which is in units of the test result. The upper and lower control limits shown on the chart are used as criteria for action. The central line represents the average or the standard value of the statistical measure being plotted.

Figure 10-1
ESSENTIALS OF A CONTROL CHART



10.2.1 Blank Spikes

Ceimic uses a measurement control program to monitor the results of laboratory preparation and analysis of control samples using statistical control charts. The analytes of interest are spiked into laboratory blank water and include the following compounds:

Semi-volatile Compounds	Graphite Furnace Metals
Phenol	As
2-Chlorophenol	Se
Acenaphthene	Pb
1,2,4-Trichlorobenzene	Tl
Volatile Compounds	**ICAP Metals**
1,1-Dichloroethene	Cu
Toluene	
Pesticide/PCB Compounds	**Cold Vapor**
Aldrin	Hg
4,4'-DDT	
	Cyanide
	Cn

The blank water spikes (lab control samples) are prepared the same time and in the same manner as project samples. It is noted directly on the control chart when a change in spike solution occurs so as to flag any recovery fluctuations as possibly reflecting the use of a new batch. Aqueous spike samples and soil spike samples for metals and cyanide are provided by the ICF branch of EPA-LV.

A control sample is introduced into each batch of water or soil samples analyzed. A batch is defined by the number of samples grouped together for QC purposes; not-to-be-exceeded control limits are based on the following calculations:

Average=$\bar{X}$=(1/n) (X_1 + X_2 + ... + Xn), where n >= 20. The estimate of the standard deviation is given by:

$$S = \{[1/(n-1)] \times \sum_{i=1} (x_i - \bar{x})^2\}^{1/2}$$

The control chart parameter estimations are as follows:

Parameter	Symbol	Formula
Centerline	CL	$\bar{X}$
Upper Control Limit	UCL	$\bar{X}$ + 3S
Lower Control Limit	LCL	$\bar{X}$ - 3S
Upper Warning Limit	UWL	$\bar{X}$ + 2S
Lower Warning Limit	LWL	$\bar{X}$ - 2S

10.2.2 Chart Analyses

If the warning limits (WL) are at the 95% level, an average of one in 20 points would exceed that limit, whereas, only one point in 100 would exceed the control limits (CL). The following actions based on these statistical criteria are required:

Control Limit

If one measurement exceeds a control limit the analysis is repeated. If the repeat analysis is

within the CL, the analysis is continued; if it exceeds the CL, the analysis is stopped and corrective action taken.

Warning Limit

If two out of three successive points exceed a WL, another sample is analyzed. If the next point is less than the WL, the analysis is continued; if the next point exceeds the WL, the analysis is stopped and corrective action taken.

Standard Deviation

If four out of five successive points exceed 1S or are in decreasing or increasing order, another sample is analyzed. If the next point is less than 1S or changes the order, the analysis is continued; if not the analysis is stopped and corrective action taken.

Central Line

If six successive samples are above the central line, another sample is analyzed. If the next point is below the central line, the analysis is continued; if the next point is on the same side, the analysis is stopped and corrective action taken.

The above considerations apply when the conditions are either above or below the central line, but not on both sides (e.g., four of five values must exceed either +1S or -1S). After the

problem is corrected, half the samples are re-analyzed between the last in-control measurement and the out-of-control one. (Standard Method of Analysis for the Examination of Water and Waste Water. 1989, 17th Edition.)

10.3 Evaluation and Criteria for Method Blanks

10.3.1 Metals

Initial and Continuing Calibration Blanks

A calibration blank is analyzed immediately after every initial and continuing calibration verification at a frequency of 10% or every two hours, whichever is more frequent. A blank is analyzed at the beginning of the run and after the last sample. If the absolute value of the blank exceeds the project-specific or the contract required detection limit (CRDL), the analysis is terminated. The analytical system is recalibrated, and all analytical samples are reanalyzed since the last acceptable calibration blank.

Preparation Blank

The preparation blank or reagent blank consists of laboratory blank water and is processed through sample preparation and analysis. Contamination of the samples is determined by the following criteria:

- If the absolute value of the blank $\leq$ the

CRDL, the sample result is not corrected.

- If the blank is > the CRDL and the sample result is < 10x the blank, the result is not corrected.
- If the blank is > the CRDL and the sample result is < 10x the blank, the samples associated with the blank are redigested and reanalyzed.
- If the blank is < the negative CRDL, all samples < 10x the CRDL are redigested and reanalyzed.

10.3.2 Organics

A method blank consists of reagent water in a volume approximate to the samples being analyzed. One blank is processed through sample preparation and analysis with each set of samples. Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are minimized. If the method blank exceeds the acceptance criteria, the analytical system is out of control and the source of the problem is investigated. Corrective action is employed and documented before analysis is continued. All samples processed with an out-of-control blank are reextracted or repurged, and reanalyzed. Acceptance criteria are below.

10.3.2.1 Volatiles Acceptance Criteria

- The method blank must contain ≤ 5x the contract required quantitation limit (CRQL) of methylene chloride, acetone, toluene, and 2-butanone.
- The method blank must contain ≤ the CRQL for all other target compounds.

10.3.2.2 Semi-volatile Acceptance Criteria

- The method blank must contain ≤ 5x the CRQL of phthalate.
- The method blank must contain ≤ the CRQL for all other target compounds.

10.3.2.3 Pesticides/PCBs Acceptance Criteria

- The method blank must contain ≤ the CRQL of every pesticide/PCB target compound.

9.0 Data Reduction, Review, and Reporting

9.1 Data Reduction

Instrumental printouts, terminal readings, chromatograms, strip chart recordings, and physical measurements provide raw analytical data that are reduced to concentrations of analytes through the application of appropriate equations. Equations are generally given within the analytical methods referenced in Section 8.0 of this Quality Assurance Plan. Data reduction may be performed manually by scientists or automatically by computerized data systems on the instruments.

9.2 Data Review

Data review is an essential element of the QA evaluation process. Review is the process of data assessment and subsequent acceptance or rejection based on established criteria. The following criteria are considered by Ceimic in the evaluation of data:

- accuracy requirements,
- precision requirements,
- detection limit requirements,
- completeness,
- representativeness,
- correctness (of manual and computer calculations),
- contractual requirements, and
- documentation requirements.

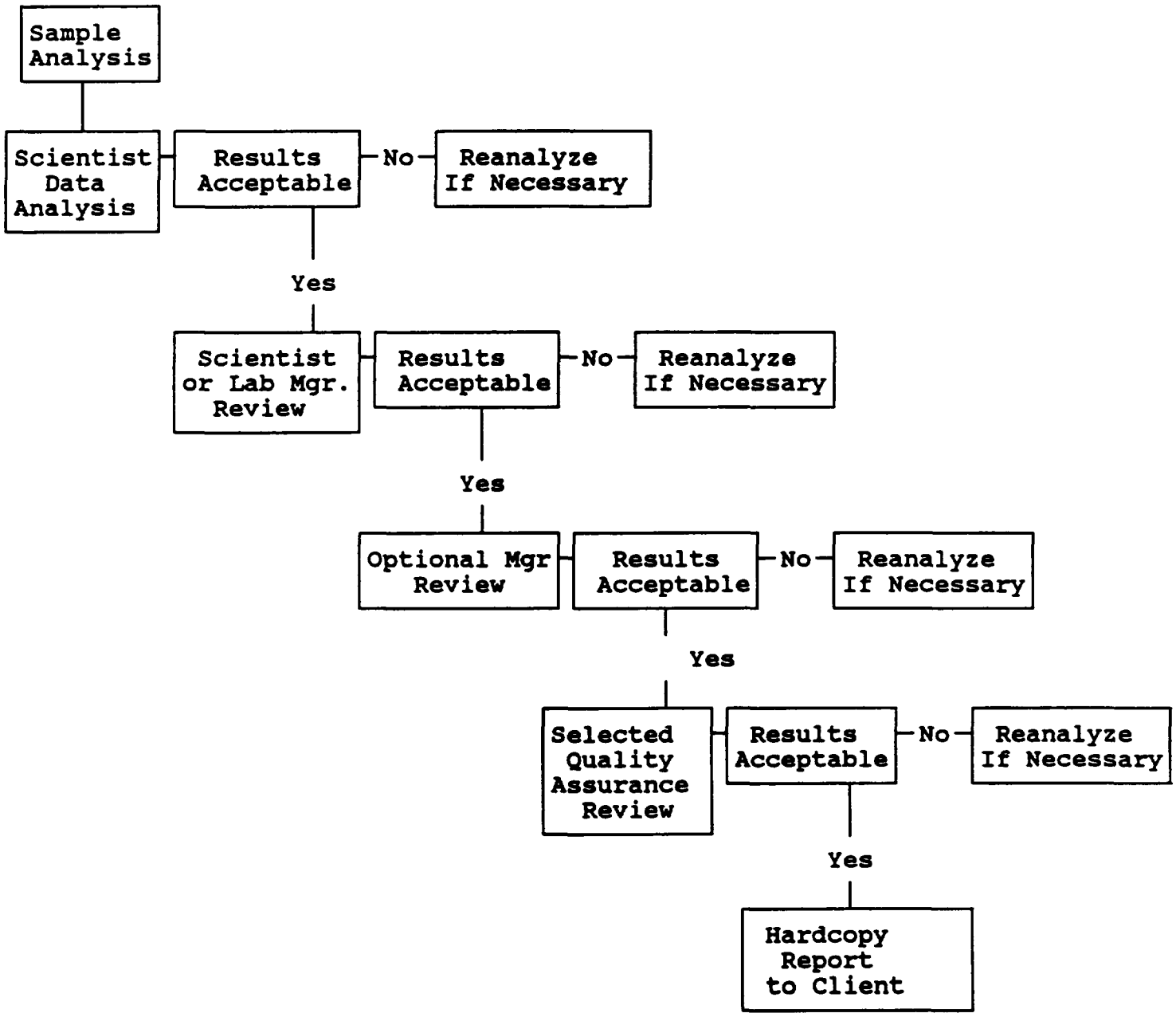
As in the case of EPA CLP procedures, data acceptance limits may be defined within the method. The same windows are used for other level 4 analyses if the sample matrix permits.

As a tracking mechanism of data acceptability, quality control charts may be plotted for specific parameters determined in identical, homogeneous matrices. Control limits for methods development and research data may be statistically determined as analytical results are generated. Review includes data assessment at both the technical and editorial levels. Technical review evaluates the application of analytical protocols and resultant effects on the data generated. Editorial review assesses the content, lucidity, conciseness, and completeness of the data report.

9.3 Data Reporting

Upon completion of data reduction and review, the scientist signs the data report form. Another scientist, experienced in the same discipline, reviews and verifies the results, also signing the data report form. The Laboratory Manager, who is responsible for the data generated in that laboratory, often performs the second tier of review or may independently review data and completed report forms. Members of the QA staff also check the results on selected sets of data. At a minimum, each data point is checked by two scientists experienced with the analytical methodology. Records are maintained for all data, even for those results that are rejected as invalid. A flow chart showing the data reduction, review, and reporting process is given in Figure 9-1.

FIGURE 9-1
DATA REDUCTION, REVIEW AND REPORTING FLOW CHART



11.0 Performance and Systems Audits

As a participant in numerous certification programs and various contracts requiring approval, Ceimic is frequently subjected to rigorous performance evaluations and on-site inspections by regulatory agencies and commercial clients. The Ceimic Quality Assurance staff performs internal audits of the laboratory as well. The audits ensure that all laboratory systems, including sample control, analytical procedures, data generation and documentation meet contractual requirements and comply with good laboratory practice standards.

11.1 Performance Audits

Ceimic realizes that accurate and reliable standards are fundamental to producing accurate and reliable results. With this in mind, Ceimic is continuously checking and rechecking the quality of its standards with the use of both external and internal performance audit samples.

The EPA CLP requires successful performance of pre-award Performance Evaluation (PE) samples prior to award of each new contract. As a member of the CLP program, our lab must continue to demonstrate performance capabilities by successfully analyzing quarterly blind PE samples.

Ceimic also participates in the Water Supply (WS) and Water Pollution (WP) Performance Evaluation Studies sponsored by the Quality Assurance Branch of the EPA. Successful analyses of these PE samples are required for laboratory certification by the environmental agencies of most states. Our laboratory also participates in the performance

evaluations of New York - both the NY ELAP and NY CLP Programs.

Performance is monitored internally on a daily basis at Ceimic through the use of surrogate standards and laboratory control samples. Check samples obtained from EPA-EMSL, Cincinnati, QA Branch, and from independent commercial sources are analyzed routinely in each of the Ceimic laboratories to ensure continuing high-level performance. In addition, Ceimic's Quality Control Department routinely audits the laboratory with the use of blind and double blind QC samples obtained from commercial vendors.

11.2 Systems Audits

The EPA, NEESA, New York DOH, New Jersey DEPE, New Hampshire DES, Rhode Island DOH and many other regulatory agencies conduct on-site audits of our laboratory for compliance with the Water Quality Act, National Primary Drinking Water Regulations and RCRA & CERCLA requirements.

The lab is audited by QA staff members in order to detect any problems with sample flow, analytical procedures or documentation and to ensure adherence to Good Laboratory Practices. The items covered in an internal systems audit may include:

- o Sample Flow
- o Chain-of-Custody
- o Sample Storage
  - Controlled access
  - Proximity to chemical storage
  - Physical conditions, e.g. temperature

- Holding times

o Sample Preparation and Analysis
  - SOPs in place
  - Logbooks
    * Standards Preparation
    * Instruments - Sample Analysis
    * Calibration/Tuning
    * Standards Analyses
    * Check Samples
    * Balance
    * Temperature
  - Notebooks
    * Dates
    * Signature
    * Filled Pages
    * Initialized and Dated Errors with Single-line Crossouts
    * Units Recorded
  - Applicable QC Samples
    * Blanks
    * Spikes
    * Duplicates
    * Surrogates
    * Control Charts
o Data File Storage
  - Hard Copies
  - Other Media - Magnetic Tape, Disk
o Container Preparation and Preservation
o Corrective Action

12.0 Preventive Maintenance and Major Instrumentation

Preventive maintenance of each analytical system is a routine practice at Ceimic. Preventive maintenance minimizes instrument downtime and consequent interruption of analysis. The laboratory instrumentation analysts are familiar with the maintenance requirements of the instruments they operate. This familiarity is based on conventional education, specialized courses, and hands-on experience. Designated staff members are trained at manufacturers' facilities in more comprehensive maintenance procedures for major analytical instrumentation. Key instrumentation is maintained under service contract. A complete listing of all major instrumentation is presented in Table 12-1.

Ceimic maintains an inventory of replacement parts required for preventive maintenance and spare parts that often need replacement, such as electron multipliers for GC/MS systems and the more mundane fuses and ferrules. In the case of a downed instrument, the problem is diagnosed as quickly as possible. If necessary, replacement parts are ordered and repairs performed by skilled in-house personnel. If necessary, a service call is placed with the manufacturer. Instrument problems and repairs are documented in logbooks kept in each laboratory.

Table 12-1

Major Laboratory Instrumentation Summary

MAJOR LABORATORY INSTRUMENTATION SUMMARY

10 Hewlett Packard Gas Chromatography/Mass Spectrometer systems with Hewlett Packard RTE 1000 Data Systems

7 Tekmar LSC 2000 Purge and Trap and ALS 2016 Autosampler Systems

21 Channels of Hewlett Packard 5890 Gas Chromatographs and Hewlett Packard RTE A-900 GC Data System

- 14 Electron Capture Detectors (ECD)
- 4 Flame Ionization Detectors (FID)
- 1 Nitrogen Phosphorus Detector (NPD)
- 1 Photo Ionization Detector (PID)
- 1 Electron Conductivity Detector (ECD)

3 Waters Gel Permeation Chromatography Systems

1 Waters HPLC system equipped with a Waters Scanning Fluorescence detector and Waters 996 Photodiode Array Detector

1 Perkin Elmer 1310 Infrared Spectrophotometer

4 Perkin Elmer 5100 Graphite Furnace Atomic Absorption Spectrometers

1 Thermo Jarrell Ash 61E Inductively Coupled Argon Plasma Spectrometer

1 Thermo Jarrell Ash 61E Trace Analyzer Inductively Coupled Argon Plasma Spectrometer

1 Perkin Elmer P-400 Inductively Coupled Argon Plasma Spectrometer

1 Leeman PS-200 Automated Mercury Analyzer

1 Dohrmann DX-20 A/B Total Organic Halide (TOX) Analyzer

1 Dohrmann DC-190 Total Organic Carbon (TOC) Analyzer

1 CEM MDS-2000 Microwave Digestion Oven

SAMPLE ANALYSIS INSTRUMENTATION DETAIL

GC/MS/DS INSTRUMENTATION

ID	Manufacturer	Model/Revision	Installation Date
GC/MS ID#1	Hewlett-Packard	5890 GC 5970 MSD	Nov. 1988
GC/MS ID#2	Hewlett-Packard	5890 GC 5970 MSD	Nov. 1988
GC/MS ID#3	Hewlett-Packard	5890 GC 5970 MSD	Nov. 1988
GC/MS ID#4	Hewlett-Packard	5890 GC 5970 MSD	Nov. 1988
GC/MS ID#5	Hewlett-Packard	5890 GC 5970 MSD	May 1990
GC/MS ID#6	Hewlett-Packard	5890 GC 5970 MSD	May 1990
GC/MS ID#7	Hewlett-Packard	5890 GC 5970 MSD	June 1991
GC/MS ID#8	Hewlett-Packard	5890 GC 5970 MSD	June 1991
GC/MS ID#9	Hewlett-Packard	5890 GC 5970 MSD	June 1991
GC/MS ID#10	Hewlett-Packard	5890 GC 5970 MSD	June 1991
Data System ID#1	Hewlett-Packard	HP 1000 RTE A	Nov. 1988
Data System ID#2	Hewlett-Packard	HP 1000 RTE A	June 1989

GC/MS/DS INSTRUMENTATION CONTINUED

ID	Manufacturer	Model/Revision	Installation Date
Data System ID#3	Hewlett-Packard	HP 1000 RTE A	May 1990
Data System ID#4	Hewlett-Packard	HP 1000 RTE A	June 1991
Data System ID#5	Hewlett-Packard	HP 1000 RTE A	June 1991
NBS Mass Spectral Library	NIH/EPA		Nov. 1988
Purge and Trap ID#1	Tekmar	LSC 2000	Nov. 1988
Autosampler	Tekmar	2016	Nov. 1988
Purge and Trap ID#2	Tekmar	LSC 2000	Nov. 1988
Autosampler	Tekmar	2016	Nov. 1988
Purge and Trap ID#3	Tekmar	LSC 2000	Nov. 1988
Autosampler	Tekmar	2016	June 1991
Purge and Trap ID#4	Tekmar	LSC 2000	May 1990
Autosampler	Tekmar	2016	May 1990

GC/MS/DS INSTRUMENTATION CONTINUED

ID	Manufacturer	Model/Revision	Installation Date
Purge and Trap ID#5	Tekmar	LSC 2000	May 1990
Autosampler	Tekmar	2016	May 1990
Purge and Trap ID#6	Tekmar	LSC 2000	June 1991
Autosampler	Tekmar	2016	June 1991
Purge and Trap ID#7	Tekmar	LSC 2000	June 1991
Autosampler	Tekmar	2016	June 1991

GC INSTRUMENTATION

ID	Manufacturer	Model	Date	Detector
GC ID#1	Hewlett-Packard	5890	Nov. 1988	ECD ECD
GC ID#2	Hewlett-Packard	5890	Nov. 1988	FID NPD
GC ID#3	Hewlett-Packard	5890	Nov. 1988	ECD ECD
GC ID#4	Hewlett-Packard	5890	Nov. 1988	PID ELCD
GC ID#5	Hewlett-Packard	5890	Sept. 1989	ECD ECD
GC ID#6	Hewlett-Packard	5890	Aug. 1989	ECD ECD
GC ID#7	Hewlett-Packard	5890	May 1990	ECD ECD
GC ID#8	Hewlett-Packard	5890	May 1990	ECD ECD
GC ID#9	Hewlett-Packard	5890	June 1991	ECD ECD
GC ID#10	Hewlett-Packard	5890	June 1991	FID FID
Data System ID#0	Hewlett-Packard	HP1000 A900 RTE A	May 1991	
Data System ID#1	Hewlett-Packard	3396B	Sept. 1988	

GC INSTRUMENTATION CONTINUED

ID	Manufacturer	Model	Installation Date
Autosampler	Hewlett-Packard	18593B	Nov. 1988
Autosampler	Hewlett-Packard	18593A	Nov. 1988
Autosampler	Hewlett-Packard	18593B	Nov. 1988
Autosampler	Hewlett-Packard	18593A	Nov. 1988
Autosampler	Hewlett-Packard	18593A	Nov. 1988
Autosampler	Hewlett-Packard	18593A	Sept. 1989
Autosampler	Hewlett-Packard	18593A	Sept. 1989
Autosampler	Hewlett-Packard	18593B	May 1990
Autosampler	Hewlett-Packard	18593B	May 1990
Autosampler	Hewlett-Packard	18593B	May 1990
Autosampler	Hewlett-Packard	18593A	May 1990
Autosampler	Hewlett-Packard	18593A	May 1990
Autosampler	Hewlett-Packard	18593B	June 1991
Autosampler	Hewlett-Packard	18593B	June 1991
Autosampler	Hewlett-Packard	18593A	June 1991

GC INSTRUMENTATION CONTINUED

ID	Manufacturer	Model	Installation Date
Data System ID#2	Hewlett-Packard	3392A	Aug. 1988
Data System ID#3	Hewlett-Packard	3396B	Nov. 1988
Data System ID#4	Hewlett-Packard	3396A	Nov. 1988
Data System ID#5	Hewlett-Packard	3396A	Nov. 1988
Data System ID#6	Hewlett-Packard	3396A	Nov. 1988
Data System ID#7	Hewlett-Packard	3396B	June 1991
Data System ID#8	Hewlett-Packard	3396B	Feb. 1992
Data System ID#9 (Prep)	Hewlett-Packard	3392A	Aug. 1988
Data System ID#10 (GPC)	Hewlett-Packard	3392A	Sept. 1988

OTHER ORGANIC ANALYSIS INSTRUMENTATION

ID	Manufacturer	Model	Installation Date
Infra-Red	Perkin-Elmer	167	Aug. 1989
Infra-Red	Perkin-Elmer	1310	Oct. 1990

OTHER ORGANIC ANALYSIS INSTRUMENTATION CONTINUED

ID	Manufacturer	Model	Installation Date
HPLC (IC)	Waters	5/10 715 ULTRA WISP RDM 484	Sept. 1990
HPLC (GPC)	Waters Fraction Collector	FRACTION	Sept. 1990
Autosampler	Waters	715	Sept. 1990
HPLC Pump	Waters	510	Sept. 1990
Tunable Absorbance Detector	Waters	484	Sept. 1990
Auto Sample Prep Station	Zymark	Benchmate	July 1992
LC Pump	SSI	300	July 1992
LC Pump	SSI	300	July 1992
LC Pump	SSI	300	July 1992
UV Detector	ISCO	UA-6	July 1992
UV Detector	ISCO	UA-6	July 1992
Fraction Collector	ISCO	Foxy 200	July 1992
Fraction Collector	ISCO	Foxy 200	July 1992

OTHER ORGANIC ANALYSIS INSTRUMENTATION CONTINUED

ID	Manufacturer	Model	Installation Date
Fraction Collector	ISCO	Foxy 200	July 1992
Total Organic Carbon Analyzer (TOC)	Dohrman	DC-190	June 1992
Total Organic Halide Analyzer (TOX)	Dohrman	DX20 A/B	July 1992

INORGANIC INSTRUMENTATION

ID	Quant.	Manufacturer	Model	Installation Date
Inductively Coupled Argon Plasma (ICAP) Spectrophotometer	1	Thermo Jarrell Ash	61E	July 1992
Autosampler for Thermo Jarrell Ash ICAP	1	Thermo Jarrell Ash	TJA-300	July 1992
Inductively Coupled Argon Plasma (ICAP) Trace Analyzer	1	Thermo Jarrell Ash	61E	July 1993
Autosampler for Thermo Jarrell Ash ICAP	1	Thermo Jarrell Ash	TJA-300	July 1993
Inductively Coupled Argon Plasma (ICAP) Spectrophotometer	1	Perkin-Elmer	P400	Oct. 1988
Autosampler for P400 ICAP	1	Perkin-Elmer	AS-51	Oct. 1988
Graphite Furnace Atomic Absorption Spectrometer	1	Perkin-Elmer	Zeeman 5100Z	Oct. 1988
Autosampler for Zeeman 5100Z	1	Perkin-Elmer	AS-60	Oct. 1988
Electrodeless Discharge Lamp Dual Power Supply	1	Perkin-Elmer	0057-0759	Oct. 1988
Graphite Furnace Atomic Absorption Spectrometer	1	Perkin-Elmer	Zeeman 5100Z	May 1990
Autosampler for Zeeman 5100Z	1	Perkin-Elmer	AS-60	May 1990

INORGANIC INSTRUMENTATION CONTINUED

ID	Quant.	Manufacturer	Model	Installation Date
Electrodeless Discharge Lamp Dual Power Supply	1	Perkin-Elmer	0057-0759	May 1990
Graphite Furnace Atomic Absorption Spectrometer	1	Perkin-Elmer	Zeeman 5100 ZL	July 1992
Autosampler for Zeeman 5100 ZL	1	Perkin-Elmer	AS-70	July 1992
Electrodeless Discharge Lamp Dual Power Supply	1	Perkin-Elmer	0303-0952	July 1992
Graphite Furnace Atomic Absorption Spectrometer	1	Perkin-Elmer	Zeeman 5100 ZL	August 1992
Autosampler for Zeeman 5100 ZL	1	Perkin-Elmer	AS-70	August 1992
Electrodeless Discharge Lamp Dual Power Supply	2	Perkin-Elmer	0303-0952	August 1992
Mercury Analyzer	2	Coleman	50B	Jan. 1989
Mercury Analyzer		Leeman Labs	PS200	Sept. 1992
Spectrophotometer	2	Spectronic 20	20	Oct. 1988
Microwave Digestion Unit	1	CEM	2000	Dec. 1991
Turbidimeter	1	HACH	18900	June 1991

INORGANIC INSTRUMENTATION CONTINUED

ID	Quant.	Manufacturer	Model	Installation Date
TCLP/Rotary Agitator	2	Associated Design	3740	Aug. 1989
TCLP/Rotary Agitator	1	Associated Design	3740	July 1990
TCLP/Rotary Agitator	1	Associated Design	3740	Nov. 1991
Recorder	1	Perkin-Elmer	56	Feb. 1989
Recorder	1	The Recorder Company	4510	Feb. 1989

OTHER INSTRUMENTATION IN LABORATORY

TCLP/Rotary Agitator	8	Associated Design	3740	Aug. 1989
TCLP/Rotary Agitator	11	Associated Design	3740	July 1990
TCLP/Rotary Agitator	11	Associated Design	3740	Nov. 1991

14.0 Corrective Action

An essential element of the QA Program, Corrective Action, provides systematic active measures to be taken in the resolution of problems and the restoration of analytical systems to proper functioning.

14.1 Laboratory Corrective Action

In the laboratory personal experience is most valuable in alerting the bench scientist to suspicious results or malfunctioning equipment. Specific QC procedures are designed to help analysts determine the need for corrective actions (see Section 9.0 of this Quality Assurance Plan, "Data Reduction, Validation, and Reporting"). Corrective actions taken by scientists in the laboratory help to avoid the production of poor quality data.

Examples of conditions that may alert the bench scientists to potential problems and warrant corrective actions are as follows:

- Tuning or calibration of instruments outside of specifications.
- QC data for precision and accuracy outside of acceptance limits.
- Undesirable trends in concentration, surrogate and spike recoveries, response factors, or relative % Difference.
- Abnormal variation in detection limits.
- Check sample results out of range.

14.2 System Corrective Action

Less obvious system problems may require more formalized, long-term corrective action. This action may be initiated by the QA Director, lab managers or bench scientists. More serious problems are brought to the attention of the lab director or president who will ensure appropriate corrective action. The essential steps in this corrective action process system are:

- Identify and define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign responsibility for implementing the corrective action.
- Implement the corrective action.
- Determine effectiveness of the corrective action. and verify it has eliminated the problem.

APPENDIX D

SPECIFICATION AND GUIDANCE FOR CONTAMINANT-FREE SAMPLE CONTAINERS

SPECIFICATIONS

AND

GUIDANCE

FOR

CONTAMINANT-FREE SAMPLE CONTAINERS

APRIL 1992

TABLE OF CONTENTS

SECTION I

INTRODUCTION

In August 1989, the Environmental Protection Agency's (EPA) Office of Emergency and Remedial Response (OERR) decentralized Superfund's Sample Container Repository program (OSWER Directive #9240.0-05). In conjunction with the decentralization of Superfund's bottle program, OERR issued specifications and guidance for preparing contaminant-free sample containers to assist the Regions in obtaining appropriate sample containers from commercially available suppliers.

The April 1992 version of "Specifications and Guidance for Contaminant-Free Sample Containers" revises the specifications and provides a single source of standardized specifications and guidance on appropriate cleaning procedures for preparing contaminant-free sample containers that meet all Contract Laboratory Program (CLP) detection/quantitation limits, including those for low concentration analyses.[1] Although the specifications and guidance procedures contained in this document are based on CLP low concentration requirements, they also are suitable for use in other analytical programs.

Specifications and guidance for preparing contaminant-free sample containers are provided in the sections that follow and are intended to describe one approach for obtaining cleaned, contaminant-free sample containers for use by groups performing sample collection activities under Superfund and other hazardous waste programs. Although other cleaning procedures may be used, sample containers must meet the criteria specified in Section II. In certain instances, the user of the sample containers may require exact adherence to the cleaning procedures and/or quality control analysis described in this document. In other instances, the user may require additional or different cleaning procedures and/or quality control analysis of the sample containers. The specific needs of the bottle user will determine the requirements for the cleaning and quality control analysis of the sample containers as long as the minimum criteria are met. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

The document has been extensively reviewed and revised since the August 1989 iteration, and important enhancements have been incorporated, including:

- Removing references to the color of the closures;
- Allowing the use of polypropylene closures as an alternative to

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[1] Because this document does not address the procurement of contaminant free sample containers, the title was changed from "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" to "Specifications and Guidance for Contaminant-Free Sample Containers."

phenolic closures;

- Referencing CLP Low Concentration Organics and Inorganics Statements of Work for the analysis of calibration verification solutions and blanks;

- Including cleaning and quality control procedures for fluoride and nitrate/nitrite;

- Removing the hexane rinse from the cleaning procedure for container types A, E, F, G, H, J, and K (semivolatile organics, pesticides, metals, cyanide, and fluoride in soils and water);

- Adding the recommendation that the bottle vendor establish and submit a Quality Assurance Plan (QAP);

- Changing the QA/QC documentation requirements so that copies of the raw data from the analyses of the QC containers are available upon request and not automatically sent to the bottle purchaser;

- Changing the permanent lot number assignment to a nine digit number from an eight digit number, where the extra digit represents the analysis parameter;

- Adding Chemical Abstract Services (CAS) registry number for the inorganic analytes in Table 1; and

- Recommending an annual demonstration of the bottle vendor's ability to meet detection limits and establish reproducibility of the cleaning techniques.

OERR and the EPA Regions decided to use the most stringent CLP requirements available to set the specifications for obtaining contaminant-free sample containers. As a result, the CLP Inorganics and Organics Low Concentration Statement of Work (SOW) requirements were selected as the basis for these specifications. Major factors in this decision included the desire to have a set of bottle cleaning specifications that met or exceeded all analytical requirements and the related need to avoid potential misuse of cleaned bottles (e.g., using a container cleaned by a multi-concentration procedure for a low concentration sample). OERR will reevaluate this decision if the low concentration requirements are deemed to be too stringent.

Most environmental sampling and analytical applications offer numerous opportunities for sample contamination. For this reason, contamination is a common source of error in environmental measurements. The sample container itself represents one such source of sample contamination. Hence, it is vital that sample containers used within the Superfund program meet strict specifications established to minimize contamination which could affect subsequent analytical determinations. Superfund sampling and analysis activities require all component materials (caps, liners, septa, packaging materials, etc.) provided by the bottle preparer to meet the criteria limits of the bottle specifications listed within Section II.

Section III provides guidance on cleaning procedures for preparing contaminant-free sample containers that meet the specifications contained in Section II. The procedures provided in this section are intended to provide sample containers that meet all current CLP Low Concentration Inorganics and Organics detection/quantitation levels.

In selecting cleaning procedures for sample containers, it is important to consider all of the parameters of interest. Although a given cleaning procedure may be effective for one parameter or type of analysis, it may be ineffective for another. When multiple determinations are performed on a single sample or on a subsample from a single container, a cleaning procedure may actually be a source of contamination for some analytes while minimizing contamination in others. It should be the responsibility of the bottle supplier to verify that the cleaning procedures actually used satisfy the quality control requirements set forth in Section IV.

Two aspects of quality assurance (i.e., quality control and quality assessment) must be applied to sample containers as well as to the analytical measurements. Quality control includes the application of good laboratory practices and standard operating procedures especially designed for the cleaning of sample containers. The cleaning operation should be based on protocols especially designed for specific contaminant problems. Strict adherence to these cleaning protocols is imperative. Quality assessment of the cleaning process depends largely on monitoring for adherence to the respective protocols. Because of their critical role in the quality assessment of the cleaning operation, protocols must be carefully designed and followed. Guidance is provided in Section IV on design and implementation of quality assurance and quality control protocols.

SECTION II

SAMPLE CONTAINER AND COMPONENT MATERIAL SPECIFICATIONS

This Section identifies sample containers commonly used in the Superfund program and provides specifications for contaminant-free sample containers for each bottle type.

A. CONTAINER MATERIAL

A variety of factors affect the choice of containers and cap material. These include resistance to breakage, size, weight, interferences with analytes of interest, cost, and availability.

Container types A through L (Figure 1, pages 7-8) are designated as the type of sample containers that have been used successfully in the past. Kimax or Pyrex brand borosilicate glass is inert to most materials and is recommended where glass containers are used (i.e., pesticides and other organics). Conventional polyethylene is recommended when plastic is acceptable because of its lower cost and lower adsorption of metal ions. The specific sampling situation will determine the use of plastic or glass.

While the sample containers shown in Figure 1 are utilized primarily for Superfund sampling activities, they also may be used for sampling activities under other programs, such as the Resource Conservation and Recovery Act (RCRA).

B. MAXIMUM CONTAMINANT LEVEL SPECIFICATIONS FOR SAMPLE CONTAINERS

The CLP, through a series of technical caucuses, has established Inorganic Contract Required Detection Limits (CRDL) and organic Contract Required Quantitation Limits (CRQL) which represent the minimum quantities needed to support the hazardous substance identification and monitoring requirements necessary for remedial and other actions at hazardous waste sites.

For inorganic sample containers, the CRDLs listed in Table 1, page 9, are the specifications for maximum trace metal contamination. Concentration at or above these limits on any parameter should preclude these containers from use in collecting inorganic samples.

The CRQL specifications for organic sample containers are listed in Table 2, pages 10-14. When the CRQL in Table 2 is multiplied by the appropriate factor listed below, the resulting value then represents the maximum concentration allowed for particular sample containers based on organic CLP sample sizes for routine analyses.

Container type	Multiple of CRQL
A	1.0
B	0.5
D	10.0
E	8.0
F	4.0
J	2.0
H	0.5
J	0.5
K	2.0

The philosophy used for determining the maximum permissible amount of contamination in a sample container was to consider the number of aliquots of sample that are available in the container and assume that the contamination present would be uniformly distributed in all of the aliquots. This assumption, and the assumption that there should be no more than one-half the CRQL contributed by the container, resulted in the establishment of contamination limits by container type. For example, the volume of container type D is sufficient to allow 20 volatile determinations. Therefore, if 10 times the CRQL of contaminant is present in the cleaned bottle, each aliquot tested will contain one-half of the CRQL of contaminant due to the contribution from the bottle.

C. GROSS CONTAMINATION

Gross contamination is defined as greater than two hundred times the acceptable concentration values in Tables 1 or 2 (multiplied by the appropriate factor), unless the cleaning procedure is successful in reducing the amount of contamination to within specifications. If this is not achieved, the grossly contaminated materials should be discarded and replaced to prevent cross contamination with other batches of containers. The bottle preparer should inspect all materials to ensure conformance with the required specifications.

FIGURE 1

SAMPLE CONTAINER
SPECIFICATIONS

Container Type	Specifications
A	Container: 80-oz amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: polypropylene or phenolic cap, 38-430 size; 0.015-in Teflon liner. Total Weight: 2.45 lbs.
B	Container: 40-mL glass vial, 24-mm neck finish. Closure: polypropylene or phenolic, open-top, screw cap, 15-cm opening, 24-400 size. Septum: 24-mm disc of 0.005-in Teflon bonded to 0.120-in silicon for total thickness of 0.125-in. Total Weight: 0.72 oz.
C	Container: 1-L high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.89 oz.
D	Container: 120-mL wide mouth, glass vial, 48-mm neck finish. Closure: polypropylene cap, 48-400 size; 0.015-in Teflon liner. Total Weight: 4.41 oz.
E	Container: 16-oz tall, wide mouth, straight-sided, flint glass jar, 63-mm neck finish. Closure: polypropylene or phenolic cap, 63-400 size; 0.015-in Teflon liner. Total Weight: 9.95 oz.
F	Container: 8-oz short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish. Closure: polypropylene or phenolic cap, 70-400 size; 0.015-in Teflon liner. Total Weight: 7.55 oz.

FIGURE 1

SAMPLE CONTAINER
SPECIFICATIONS
(Continued)

Container Type	Specifications
G	Container: 4-oz tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish. Closure: polypropylene or phenolic cap, 48-400 size; 0.015-in Teflon liner. Total Weight: 4.70 oz.
H	Container: 1-L amber, Boston round, glass bottle, 33-mm pour-out neck finish. Closure: polypropylene or phenolic cap, 33-430 size; 0.015-in Teflon liner. Total Weight: 1.11 lbs.
J	Container: 32-oz tall, wide mouth, straight-sided, flint glass jar, 89-mm neck finish. Closure: polypropylene or phenolic cap, 89-400 size; 0.015-in Teflon liner. Total Weight: 1.06 lbs.
K	Container: 4-L amber glass, ring handle bottle/jug, 38-mm neck finish. Closure: polypropylene or phenolic cap, 38-430 size; 0.015-in Teflon liner. Total Weight: 2.88 lbs.
L	Container: 500-mL high-density polyethylene, cylinder-round bottle, 28-mm neck finish. Closure: polypropylene cap, ribbed, 28-410 size; F217 polyethylene liner. Total Weight: 1.20 oz.

TABLE 1

INORGANIC ANALYTE
SPECIFICATIONS

	Analyte	CAS Number	Contract Required Detection Limits* (µg/L)
1.	Aluminum	7429-90-5	100
2.	Antimony	7440-36-0	5
3.	Arsenic	7440-38-2	2
4.	Barium	7440-39-3	20
5.	Beryllium	7440-41-7	1
6.	Cadmium	7440-43-9	1
7.	Calcium	7440-70-2	500
8.	Chromium	7440-47-3	10
9.	Cobalt	7440-48-4	10
10.	Copper	7440-50-8	10
11.	Iron	7439-89-6	500
12.	Lead	7439-92-1	2
13.	Magnesium	7439-95-4	500
14.	Manganese	7439-96-5	10
15.	Mercury	7439-97-6	0.2
16.	Nickel	7440-02-0	20
17.	Potassium	7440-09-7	750
18.	Selenium	7782-49-2	3
19.	Silver	7440-22-4	10
20.	Sodium	7440-23-5	500
21.	Thallium	7440-28-0	10
22.	Vanadium	7440-62-2	10
23.	Zinc	7440-66-6	20
24.	Cyanide	57-12-5	10
25.	Fluoride	16984-48-8	200
26.	Nitrate/Nitrite	1-00-5	100

*CRDLs are based on the CLP Inorganics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS

	Volatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
1.	Chloromethane	74-87-3	1
2.	Bromomethane	74-83-9	1
3.	Vinyl Chloride	75-01-4	1
4.	Chloroethane	75-00-3	1
5.	Methylene Chloride	75-09-2	2
6.	Acetone	67-64-1	5
7.	Carbon Disulfide	75-15-0	1
8.	1,1-Dichloroethene	75-35-4	1
9.	1,1-Dichloroethane	75-34-3	1
10.	cis-1,2-Dichloroethene	156-59-4	1
11.	trans-1,2-Dichloroethene	156-60-5	1
12.	Chloroform	67-66-3	1
13.	1,2-Dichloroethane	107-06-2	1
14.	2-Butanone	78-93-3	5
15.	Bromochloromethane	74-97-5	1
16.	1,1,1-Trichloroethane	71-55-6	1
17.	Carbon Tetrachloride	56-23-5	1
18.	Bromodichloromethane	75-27-4	1
19.	1,2-Dichloropropane	78-87-5	1
20.	cis-1,3-Dichloropropene	10061-01-5	1
21.	Trichloroethene	79-01-6	1
22.	Dibromochloromethane	124-48-1	1
23.	1,1,2-Trichloroethane	79-00-5	1
24.	Benzene	71-43-2	1
25.	trans-1,3-Dichloropropene	10061-02-6	1
26.	Bromoform	75-25-2	1
27.	4-Methyl-2-pentanone	108-10-1	5
28.	2-Hexanone	591-78-6	5
29.	Tetrachloroethene	127-18-4	1
30.	1,1,2,2-Tetrachloroethane	79-34-5	1

[1]CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Volatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31.	1,2-Dibromoethane	106-93-4	1
32.	Toluene	108-88-3	1
33.	Chlorobenzene	108-90-7	1
34.	Ethylbenzene	100-41-4	1
35.	Styrene	100-42-5	1
36.	Xylenes (total)	1330-20-7	1
37.	1,3-Dichlorobenzene	541-73-1	1
38.	1,4-Dichlorobenzene	106-46-7	1
39.	1,2-Dichlorobenzene	95-50-1	1
40.	1,2-Dibromo-3-chloropropane	96-12-8	1

[1]CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits* (µg/L)
1.	Phenol	108-95-2	5
2.	bis-(2-Chloroethyl)ether	111-44-4	5
3.	2-Chlorophenol	95-57-8	5
4.	2-Methylphenol	95-48-7	5
5.	2,2'-oxybis-(1-Chloropropane)	108-60-1	5
6.	4-Methylphenol	106-44-5	5
7.	N-Nitroso-di-n-dipropylamine	621-64-7	5
8.	Hexachloroethane	67-72-1	5
9.	Nitrobenzene	98-95-3	5
10.	Isophorone	78-59-1	5
11.	2-Nitrophenol	88-75-5	5
12.	2,4-Dimethylphenol	105-67-9	5
13.	bis-(2-Chloroethoxy)methane	111-91-1	5
14.	2,4-Dichlorophenol	120-83-2	5
15.	1,2,4-Trichlorobenzene	120-82-1	5
16.	Naphthalene	91-20-3	5
17.	4-Chloroaniline	106-47-8	5
18	Hexachlorobutadiene	87-68-3	5
19.	4-Chloro-3-methylphenol	59-50-7	5
20.	2-Methylnaphthalene	91-57-6	5
21.	Hexachlorocyclopentadiene	77-47-4	5
22.	2,4,6-Trichlorophenol	88-06-2	5
23.	2,4,5-Trichlorophenol	95-95-4	20
24.	2-Chloronaphthalene	91-58-7	5
25.	2-Nitroaniline	88-74-4	20
26.	Dimethylphthalate	131-11-3	5
27.	Acenapnthylene	208-96-8	5
28.	2,6-Dinitrotoluene	606-20-2	5
29.	3-Nitroaniline	99-09-2	20
30.	Acenaphthene	83-32-9	5

*CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Semivolatiles	CAS Number	Contract Required Quantitation Limits[1] (µg/L)
31.	2,4-Dinitrophenol	51-28-5	20
32.	4-Nitrophenol	100-02-7	20
33.	Dibenzofuran	132-64-9	5
34.	2,4-Dinitrotoluene	121-14-2	5
35.	Diethylphthalate	84-66-2	5
36.	4-Chlorophenyl-phenylether	7005-72-3	5
37.	Fluorene	86-73-7	5
38.	4-Nitroaniline	100-01-6	20
39.	4,6-Dinitro-2-methylphenol	534-52-1	20
40.	N-Nitrosodiphenylamine	86-30-6	5
41.	4-Bromophenyl-phenylether	101-55-3	5
42.	Hexachlorobenzene	118-74-1	5
43.	Pentachlorophenol	87-86-5	20
44.	Phenanthrene	85-01-8	5
45.	Anthracene	120-12-7	5
46.	Di-n-butylphthalate	84-74-2	5
47.	Fluoranthene	206-44-0	5
48	Pyrene	129-00-0	5
49.	Butylbenzylphthalate	85-68-7	5
50.	3,3'-Dichlorobenzidine	91-94-1	5
51.	Benz[a]anthracene	56-55-3	5
52.	Chrysene	218-01-9	5
53.	bis-(2-Ethylhexyl)phthalate	117-81-7	5
54.	Di-n-octylphthalate	117-84-0	5
55.	Benzo[b]fluoranthene	205-99-2	5
56.	Benzo[k]fluoranthene	207-08-9	5
57.	Benzo[a]pyrene	50-32-8	5
58.	Indeno(1,2,3-cd)pyrene	193-39-5	5
59.	Dibenz[a,h]anthracene	53-70-3	5
60.	Benzo[g,h,i]perylene	191-24-2	5

[1]CRQLs are based on the CLP Organics Low Concentration SOW

TABLE 2

ORGANIC COMPOUND
SPECIFICATIONS
(Continued)

	Pesticides/PCBs	CAS Number	Contract Required Quantitation Limits* (µg/L)
1.	alpha-BHC	319-84-6	0.01
2.	beta-BHC	319-85-7	0.01
3.	delta-BHC	319-86-8	0.01
4.	gamma-BHC (Lindane)	58-89-9	0.01
5.	Heptachlor	76-44-8	0.01
6.	Aldrin	309-00-2	0.01
7.	Heptachlor epoxide	1024-57-3	0.01
8.	Endosulfan I	959-98-8	0.01
9.	Dieldrin	60-57-1	0.02
10.	4,4'-DDE	72-55-9	0.02
11.	Endrin	72-20-8	0.02
12.	Endosulfan II	33213-65-9	0.02
13.	4,4'-DDD	72-54-8	0.02
14.	Endosulfan sulfate	1031-07-8	0.02
15.	4,4'-DDT	50-29-3	0.02
16.	Methoxychlor	72-43-5	0.10
17.	Endrin ketone	53494-70-5	0.02
18	Endrin aldehyde	7421-36-3	0.02
19.	alpha-Chlordane	5103-71-9	0.01
20.	gamma-Chlordane	5103-74-2	0.01
21.	Toxaphene	8001-35-2	1.0
22.	Aroclor-1016	12674-11-2	0.20
23.	Aroclor-1221	11104-28-2	0.20
24.	Aroclor-1232	11141-16-5	0.40
25.	Aroclor-1242	53469-21-9	0.20
26.	Aroclor-1248	12672-29-6	0.20
27.	Aroclor-1254	11097-69-1	0.20
28.	Aroclor-1260	11096-82-5	0.20

*CRQLs are based on the CLP Organics Low Concentration SOW

c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

d. Rinse three times with ASTM Type I deionized water.

e. Invert and air dry in a contaminant-free environment.

f. Cap bottles.

g. Label each container with the lot number and pack in a case.

h. Label exterior of each case with the lot number.

i. Store in a contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step C.1.c.

b. Follow all other steps in the cleaning procedure described in part C.1 above.

SECTION III

SAMPLE CONTAINER PREPARATION AND CLEANING PROCEDURES

This Section is provided as guidance for the preparation of sample containers that meet the contaminant-free specifications contained in Section II. There are various procedures for cleaning sample containers depending upon the analyses to be performed on the sample. The following cleaning procedures are modeled after those specified for the Superfund Sample Container Repository program. Other suitable cleaning procedures exist and may be used as long as the sample containers meet the criteria established in Section II. In some instances, the specific needs of the bottle user may dictate exact adherence to the sample container preparation and cleaning procedures that follow; while in other instances, modifications may be required. It is the responsibility of the bottle user to define the sample container preparation, cleaning, and quality control requirements.

A. Cleaning Procedure for Container Types: A, E, F, G, H, J, and K

1. Sample Type: Semivolatile Organics, Pesticides, Metals, Cyanide, and Fluoride in Soils and Water.

   a. Wash glass bottles, Teflon liners, and caps with hot tap water using laboratory grade nonphosphate detergent.

   b. Rinse three times with copious amounts of tap water to remove detergent.

   c. Rinse with 1:1 nitric acid (reagent grade HNO_3, diluted with ASTM Type I deionized water).

   d. Rinse three times with ASTM Type I organic free water.

   e. Oven dry bottles, liners, and caps at 105-125°C for one hour.

   f. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   g. Rinse bottles with pesticide grade methylene chloride (or other suitable solvents specified by the bottle user) using 20 mL for 4-gallon containers; 10 mL for 32-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.

   h. Oven dry bottles, liners, and caps at 105-125°C for one hour.

   i. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.

   j. Place liners in lids and cap containers.

k. Label each container with the lot number and pack in a case.

l. Label exterior of each case with the lot number.

m. Store in a contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.

a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step A.1.c.

b. Follow all other steps in the cleaning procedure described in part A.1 above.

B. Cleaning Procedure for Container Types: B, D

1. Sample Type: Purgeable (Volatile) Organics in Soils and Water.

a. Wash glass vials, Teflon-backed septa, Teflon liners, and caps in hot water using laboratory grade nonphosphate detergent.

b. Rinse three times with copious amounts of tap water to remove detergent.

c. Rinse three times with ASTM Type I organic-free water.

d. Oven dry vials, caps, septa, and liners at 105-125°C for one hour.

e. Allow vials, caps, septa, and liners to cool to room temperature in an enclosed contaminant-free environment.

f. Seal 40-mL vials with septa (Teflon side down) and cap.

g. Place liners in lids and cap 120-mL vials.

h. Label each vial with the lot number and pack in a case.

i. Label exterior of each case with the lot number.

j. Store in a contaminant-free area.

C. Cleaning Procedure for Container Types: C, L

1. Sample Type: Metals, Cyanide, and Fluoride in Soils and Water.

a. Wash polyethylene bottles and caps in hot tap water using laboratory-grade nonphosphate detergent.

b. Rinse three times with copious amounts of tap water to remove detergent.

SECTION IV

SAMPLE CONTAINER QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

A. Quality Assurance

The objectives of this Section are to: (1) present procedures for evaluating quality assurance (QA) information to ensure that specifications identified in Section II have been met; and (2) discuss techniques for the quality control (QC) analysis of sample containers to be used in conjunction with the cleaning procedures contained in Section III.

The bottle vendor should establish a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements, production procedures, and tracking systems. The QAP should incorporate procedures for the inspection of incoming raw materials; preparation, cleaning, and labeling of container lots; quality control analyses of cleaned container lots; document control, including all documentation required for analysis, packing, shipping, and tracking of container lots; any necessary corrective actions; and any quality assessment measures implemented by management to ensure acceptable performance. The QAP should be available and provided to the bottle purchaser upon request.

Major QA/QC activities should include the inspection of all incoming materials, QC analysis of cleaned lots of containers, and monitoring of the container storage area. Complete documentation of all QC inspection results (acknowledging acceptance or rejection) should be kept as part of the permanent bottle preparation files. QA/QC records (e.g., preparation/QC logs, analytical data, data tapes, storage log) also should be stored in a central location within the facility.

Documentation indicating that the container lot has passed all QA/QC requirements should be provided by the bottle vendor to the bottle purchaser with each container lot. Documentation should include a signed and dated cover statement affirming that all QA/QC criteria were met. Copies of raw data from applicable analyses of the QC containers, laboratory standards, check samples, and blanks should be available and provided upon request. Original documentation should be retained for at least 10 years. Minimum documentation that should be available, if applicable, for each lot of containers includes:

- A statement that "Sample container lot _____ meets or exceeds all QA/QC criteria established in 'Specifications and Guidance for Contaminant-Free Sample Containers;'"
- Reconstructed Ion Chromatographs (RICs) from volatile and semivolatile organics determinations, including calibration verification standards, check samples, and blanks;
- GC chromatographs from pesticides determinations, including calibration verification standards, check samples, and blanks;

- ICP, hydride-ICP, or ICP-MS instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks;

- AA raw data sheets and instrument readouts from metals determinations, including calibration verification standards, check samples, and blanks; and

- Cyanide, fluoride, and nitrate/nitrite raw data sheets and instrument readouts from these determinations, including calibration verification standards, check samples, and blanks.

Prior to the first shipment of containers, and at least annually thereafter, the bottle vendor should demonstrate its ability to meet the CRDLs and CRQLs, and establish the reproducibility of the cleaning techniques for each bottle type. The ability to meet the CRDLs and CRQLs is accomplished through the determination of instrument detection limits (IDLs). The bottle vendor should use the procedures in the current CLP Low Concentration Inorganics and Organics SOWs to determine IDLs. IDLs should be below the CRDLs or CRQLs. To establish the reproducibility for each bottle type, the bottle vendor should randomly pick seven containers from a cleaned lot and analyze as described in the Quality Control Analysis part of this Section. Parameter concentrations should be at or below the CRDL or CRQL for each bottle type. Documentation from these analyses should be available and provided upon request.

1. Incoming Materials Inspection:

A representative item from each case of containers should be checked for conformance with specifications provided in Section II. Any deviation should be considered unacceptable. A log of incoming shipments should be maintained to identify material type, purchase order number, and delivery date. The date of incoming inspection and acceptance or rejection of the material should also be recorded on this log.

2. Quality Control Inspection of Cleaned Lots of Containers:

Following container cleaning and labeling, containers should be randomly selected from each container lot to be used for QC purposes. The two categories of QC containers should be as follows:

a. Analysis QC Containers:

One percent of the total number of containers in each lot should be designated as the analysis QC container(s). For lots of less than 100 containers, one container should be designated as the analysis QC container. The sample container preparer should analyze the analysis QC container(s) to check for contamination prior to releasing the associated container lot for shipment. The QC analyses procedures specified in the Quality Control Analysis part of this Section for determining the presence of semivolatile and volatile organics, pesticides, metals, cyanide, fluoride, and nitrate/nitrite should be utilized.

For each analysis QC container(s), an appropriate QC number should be assigned that cross-references the QC container to the related lot of containers. For example, the QC number could be a seven-digit number sequentially assigned to each lot that has undergone QC analysis. Under this numbering scheme, the first alphabetical character would be the container type letter from Figure 1, the next four digits would be assigned sequentially in numerical order starting with "0001" for the first lot to undergo QC analyses, the sixth character would indicate the number of QC container for the lot, (e.g., "1" for the first QC container in the lot, "2" for the second, etc.) and the last character would be either a "C" to indicate clearance or an "R" to indicate rejection.

If the representative analysis QC container(s) passes QC inspection, the related lot of containers should be released, and the appropriate QC number should be entered in the preparation/QC log to indicate clearance of the lot for shipment.

If the analysis QC container(s) are found to be contaminated per the specified QC analysis procedures, the appropriate QC rejection number should be assigned and entered in the preparation/QC log. Any container labels should be removed and the entire lot returned for reprocessing under a new lot number. Excessive QC rejection for a particular container type should be noted for future reference.

A laboratory standard, check sample, and a blank should be run with each QC analysis. A calibration verification standard should be analyzed once every 12 hours. All QC analysis results should be kept in chronological order by QC report number in a central QC file. The QC numbers assigned should be documented in the preparation/QC log, indicating acceptance or rejection and date of analysis.

A container lot should not be released for shipment prior to QC analysis and clearance. Once the containers have passed QC inspection, the containers should be stored in a contaminant-free area until packaging and shipment.

b. Storage QC Containers:

One QC container per lot should be designated as the storage QC container. The storage QC container should be separated from the lot after cleaning and labeling and should be stored in a designated contaminant-free area for one year. The date the container is placed in the storage area should be recorded in the storage QC container log.

If contamination of the particular container lot comes into question at any time following shipment, the storage QC container should be removed from the storage area and analyzed using the QC analysis procedures for that container type (see Quality Control Analysis, this Section). Upon removal, containers should be logged out of the storage area.

The designated storage area should be monitored continuously for volatile contaminants in the following manner. A precleaned, 40-mL vi

that has passed a QC inspection should be filled with ASTM Type I organic-free water and be placed in the storage area. This vial should be changed at one-week intervals. The removed vial should be subjected to analysis for volatile organics as described in the Quality Control Analysis part of this Section. Any peaks indicate contamination. Identify contaminants, if present, and include the results in a report to all clients who purchased bottles from the affected lot(s).

3. Quality Control Analysis

The types of QC analyses correlate with the types of containers being analyzed and their future use in sample collection. The QC analyses are intended for the determination of:

- Semivolatile organics and pesticides;
- Volatile organics;
- Metals;
- Cyanide;
- Fluoride; and
- Nitrate/Nitrite.

QC analyses should be performed according to the container type and related sample type and utilize the specific method(s) described below.

1. Determination of Semivolatile Organics and Pesticides:

Container Types: A, E, F, G, H, J, and K

a. Sample Preparation:

- Add 60 mL of pesticide-grade methylene chloride to the container and shake for two minutes.
- Transfer the solvent to a Kuderna-Danish (KD) apparatus equipped with a three-ball Snyder column. Concentrate to less than 10 mL on a steam bath. Split the solvent into two 5 mL fractions for semivolatile and pesticide determinations.
- Add 50 mL of pesticide-grade hexane (for pesticide determinations only) to the KD apparatus by slowly pouring down through the Snyder column. Concentrate to less than 10 mL to effect solvent replacement of hexane for methylene chloride.
- Concentrate the solvent to 1 mL using a micro-Snyder column.
- Prepare a solvent blank by adding 60 mL of the rinse solvent used in step "g" of the cleaning procedure for container types A, E.

F, G, H, J, and K (Section III page 15) directly to a KD apparatus, and proceed as above.

b. Semivolatile Organics Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:

  (1) If problems are encountered meeting the %RSD criteria on the initial calibration for semivolatiles, the high concentration point should be deleted and a four-point calibration used.

  (2) The low concentration standard should be used for the continuing calibration standard for semivolatile analyses.

  (3) The percent difference window should be widened to ± 30 percent for all compounds.

- Inject 1 µL of solvent into a gas chromatograph/mass spectrometer (GC/MS).

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.

- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the nine semivolatile organic compounds listed in Table 3 (page 29) with concentrations in the 5-20 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

c. Pesticides Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW.

- Inject 1 µL of solvent into a gas chromatograph (GC) equipped with an electron capture detector (ECD).

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW.

- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the seven pesticide compounds listed in Table 3 (page 29) with concentrations in the 0.01 to 1 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

2. Determination of Volatile Organics:

Container Types: S and D

a. Sample Preparation:

- Fill the container with ASTM Type I organic-free water.

- Cap the container and let stand for 48 hours.

b. Sample Analysis:

- Instrument calibration should be performed as described in the most recent CLP Low Concentration Organics SOW with the following exceptions:

  (1) If problems are encountered meeting the %RSD criteria on the initial calibration for volatiles, the high concentration point should be deleted and a four-point calibration used.

  (2) The low concentration standard should be used for the continuing calibration standard for volatile analyses.

  (3) The percent difference window should be widened to ± 30 percent.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Organics SOW

- Blanks should be run as described in the most recent CLP Low Concentration Organics SOW. The blank should consist of an aliquot of the ASTM Type I water used in the sample preparation

- If peaks are found in the container blank that are not in the solvent blank, or if the container blank peak heights or areas

are greater than 50 percent of the solvent blank peak heights or areas, the containers should be rejected.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.

- A standard mixture of the five volatile organic compounds listed in Table 3 (page 29) with concentrations in the 1-5 ppb range should be analyzed to ensure that sensitivities are achieved to will meet contract required quantitation limits. This standard should be prepared from a different source from the calibration standards.

3. Determination of Metals:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Add 100 mL of ASTM Type I deionized water to the container, and acidify with 1.0 mL of reagent-grade HNO_3. Cap and shake for three to five minutes.

- Cap the container and let stand for 48 hours.

- Treat the sample as a dissolved metals sample. Analyze the undigested water using the most recent CLP Low Concentration Inorganics SOW.

b. Sample Analysis:

- Instruments used for the analysis of the samples should meet the contract required detection limits in Table 1.

- The ASTM Type I deionized water should be analyzed before use on the bottles that are designated for analysis to ensure that contaminated water is not used for rinsing the bottles.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW. A calibration blank is a solution made up exactly like the sample preparation solution. The calibration blank should be less than the values contained in Table 1.

- A set of standards in the expected working range should be analyzed with each analytical run. The acid matrix of the standards, blank, and quality control samples should match that of the samples.

- Concentrations at or above the detection limit for each parameter (listed in Table 1) should be cause for rejection of the lot of containers. **NOTE:** The sodium detection limit for container types A, E, F, G, H, J, and K is 5000 µg/L unless the containers will be used for low concentration analyses, then the detection limit is 500 µg/L.

4. Determination of Cyanide:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container. Add 1.25 mL of 6N NaOH (for container types F and G use 100 mL of ASTM Type I deionized water and 0.5 mL of 6N NaOH). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 10 µg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 10 µg/L cyanide (or greater) should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container, the cap, or the NaOH.

5. Determination of Fluoride:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 200 µg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a check sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 200 µg/L (or greater) of fluoride should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

6. Determination of Nitrate/Nitrite:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL of ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the most recent CLP Low Concentration Inorganics SOW.

- The detection limit should be 100 µg/L or lower.

- Calibration verification standards should be analyzed as described in the most recent CLP Low Concentration Inorganics SOW.

- Blanks should be run as described in the most recent CLP Low Concentration Inorganics SOW. The calibration blank should consist of an aliquot of the ASTM Type I water used above.

- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample was prepared.

- The detection of 100 µg/L (or greater) of nitrate/nitrite should be cause for rejection of the lot of containers. **NOTE:** Contamination could be due to the container or the cap.

C. Preparation and Labeling

Sampling for environmental specimens requires that sample containers be transported to field sites prior to sample collection. As a result, considerable time may elapse between the receipt of sample containers and collection of the samples. Because of the large number of samples taken at any one site, accounting for all sample containers can become extremely difficult. The following guidance on the identification and tracking of sample containers is based on procedures that have been used successfully in the CLP bottle program.

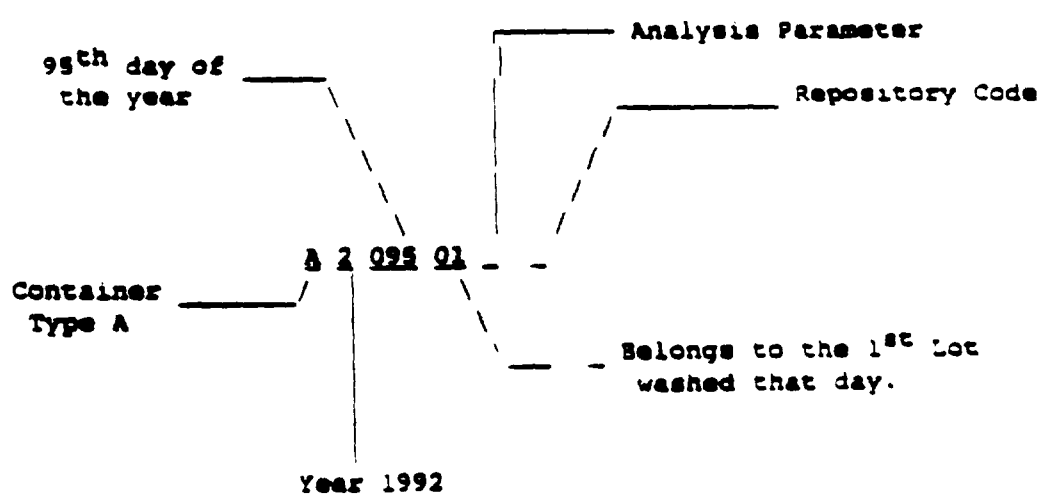
1. Each shipment should be inspected to verify that the requested number of cleaned and prepared sample containers have been supplied and meet the requirements specified in Section II (Tables 1 and 2). If any shipment fails to meet the required specifications, it should be discarded and replaced with a supply of sample containers that meet the required criteria.

2. The sample containers should be removed and prepared in accordance with the methods designated below.

3. A permanent nine-digit lot number should be assigned to each lot of sample containers for identification and tracking purposes throughout the life of the containers. Figure 2 provides an example of a lot number sequence.

FIGURE 2

LOT NUMBER SEQUENCE



a. The first digit represents the container type in Section II (Figure 1).

b. The second digit represents the last digit of the calendar year.

c. The next three digits represents the day of the year on which the sample containers were washed.

d. The sixth and seventh digits represent the daily lot number.

e. The eighth digit represents the analysis parameter where:

A = Semivolatile organics, pesticides, metals, cyanide, and fluoride;
B = Metals, cyanide, and fluoride;
V = Volatile organics;
S = Semivolatile organics and/or pesticides;
M = Metals;
C = Cyanide;
F = Fluoride; and
N = Nitrate/nitrite.

f. The final digit represents the identification of the person who prepared the lot.

4. The lot number for each container should be entered, along with the date of washing, type of container, and number of containers per lot, into the preparation/QC log book.

5. Lot numbers printed with solvent resistant ink on a nonremovable label should remain with the corresponding containers throughout the cleaning procedure.

6. After sample container cleaning and drying, the label should be affixed to the containers in a permanent manner.

7. At least one face should be clearly marked, excluding the top and bottom faces, of each case of sample containers with the assigned lot numbers.

TABLE 3

STANDARD MIXTURES OF ORGANIC COMPOUNDS TO VERIFY SENSITIVITY

Volatiles	Semivolatiles	Pesticides
Methylene Chloride	Nitrobenzene	Gamma-BHC
Acetone	4-Chloroaniline	Heptachlor
2-Butanone	2,6-Dinitrotoluene	Aldrin
Trichloroethene	Diethylphthalate	Dieldrin
Toluene	4-Bromophenyl-phenylether	Endrin
	Hexachlorobenzene	4,4'-DDT
	Pentachlorophenol	Aroclor 1260
	Di-n-butylphthalate	
	bis(2-Ethylhexyl)phthalate	